

Date: August 31 1991

AD-A246 511



AUG 30 1991

FINAL TECHNICAL REPORT

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For: US Naval Research Laboratory, Project N-00014-90-P-2030

"Characterization of Semi-Insulating Gallium Arsenide"

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Abstract:

for the purpose of
This project was established effective March 9 1990, to continue through August 31 1991. Its purpose has been characterization of samples from melt-grown single crystals of nominally undoped GaAs, primarily such crystals as grown by the vertical zone melt (VZM) method at the Naval Research Laboratory. During this period of 18 months, electrical and optical samples have been measured for 13 of these VZM-grown GaAs crystals. Our collaborators at NRL have received technical information on these results and their interpretation for 11 of the VZM crystals in technical and quarterly reports of various dates during the period. (They have also received information from us concerning samples from three crystals grown in Japan by Nippon Mining Co. by the Czochralski method.) Results specific for the final two NRL-grown VZM crystals of this project are included in the present Final Report.

Measurements made at Western Washington University (WWU) under the terms of this project have been in accordance with the Statement of Work provided at the project's outset: including near-IR transmittance measurements for determination of neutral and ionized EL2 center concentrations, local vibrational mode (LVM) measurements in the mid-IR relative to carbon impurities, and measurements of dc electrical transport properties versus temperature. Results provided herein for Crystals 4-88H and 27-19N typify the methodology which has been developed and used during the course of the project.

The measurements made at WWU have complemented the work at NRL itself, of experimental adjustment of the parameters for crystal growth and thermal history, and of microscopic studies of dislocations and other defect structures.

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Introduction

→ A program for growth of GaAs and other semiconductors by the VZM method began at NRL several years ago, and the practicality of this method for growing semi-insulating (SI) nominally undoped GaAs in a pBN crucible was demonstrated by Swiggard (1989) and in various subsequent papers. The writer of this report (JSB) is glad to have been able to collaborate in characterization of these SI VZM GaAs crystals since those early days. Our characterization work at WWU since March 1990 has been supported in part by the NRL-funded project for which this is the final technical report.

As indicated in Table I on page 3, we have been involved in characterization of samples from 13 of these NRL-grown VZM crystals, involving 26 optical samples, and 27 electrical samples. Further on in this report are our actual findings for the last eight of these samples: four optical samples and four electrical ones from Crystals 4-88H, and 27-19N. (A brief synopsis of the room temperature data for Crystal 4-88H was provided previously, on 8/16/91.)

In addition to the various samples from these 13 NRL-grown VZM crystals, an opportunity arose during the project period to examine undoped SI GaAs grown in Japan by the LEC method, crystals grown at the R&D Center of Nippon Mining Co. Samples were electrically and optically measured from three such LEC crystals, each having received a different mode of post-growth thermal anneal sequencing. Since the thermal history of an undoped GaAs crystal affects its behavior, in addition to the thermal conditions (thermal gradient, etc.) during growth, there appeared to be value in making this investigation of the Nippon samples, and in reporting what we found to our NRL colleagues. This was done in two reports, dated March 18 and March 26 1991. We were also able to provide Drs. Henry and Nordquist of NRL at that time with copies of the "Specification Sheets" showing the results of measurements made in Japan on material from those three crystals.

Before getting down to the specifics of Crystals 4-88H and 27-19N, it is appropriate first to comment briefly (and illustrate) the types of measurement we have made which are - in the writer's view - especially useful for GaAs whose low conductivity places it in the SI range.

Electrical Measurements

The measurement of dc electrical resistivity and of the dc Hall coefficient are about the most basic quantities one can attempt to evaluate for a nonmetal. The van der Pauw (vdP) arrangement of four contacts about the periphery of a flat plate sample (the four corners of a square are admirable, and are what we strive for) makes it feasible in principle to deduce resistivity ρ from just two 4-wire measurements, though we make eight in order to permute contacts, reverse current, etc. The Hall coefficient R_H can also be derived with just two 4-wire readings (reversing only the magnetic field direction), though once again we make eight observations with current reversals and contact permutations. All of the above are quite straightforward for a sample for which ρ lies in the range from (say) 0.1 ohm cm to (say) 500 ohm cm. There are troubles (which do not need to concern us at the present) for lower resistivity, since measurement voltages become rather small. There are other troubles for high resistivity materials (SI GaAs par excellence) because of the small controlled currents needed, leakage effects, RC time constants, long acquisition time, etc.

For the above reasons we have developed and use a guarded cable approach with FET OpAmps to make computer-driven dc parameter acquisition reliable for sample impedance of 10^{10} ohms or more, and with all the 16 observations made

[Text resumes on page 4]

Table I. VZM-Grown Crystals from NRL for which Optical and Electrical Samples were measured in this Project at WWU.

NRL Crystal Number	Optical Samples Measured by us	Electrical Samples Measured by us	Period When these Samples Measured
25-66N	II-7 III-00	II-2 III-1	March 1990
25-69N	II-6 III-6	II-5 III-6 IV-1	March 1990
26-5N	II-1 III-1	II-2 III-2	July 1990
26-11N	II-1 III-1 IV-1	II-2 III-2 IV-2	Aug-Oct. 1990
26-16N	II-1	II-4	Sept-Nov. 1990
26-41N	II-1 III-1	II-8 III-8	Feb-April 1991
26-52N	II-1 III-1	II-3 III-8	March-April 1991
26-90N	II-1 III-9	II-4 III-3	May 1991
27-1N	II-1 III-1	II-4 III-2	May-June 1991
27-5N	II-1 III-1	II-5 III-3	June-July 1991
27-12N	II-1 II-36	II-5 II-31	July 1991
4-88H	II-1 III-16	II-7 III-9	August 1991
27-19N	II-1 II-31	II-5 II-25	August 1991

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in the shortest possible time (2 minutes maximum) so as to minimize any possible temperature variation during the 16-observation sequence. The latter is highly important, since a 0.1°C temperature change affects ρ and the electron concentration n_o in SI GaAs by about 1%.

In addition to making reliable dc measurements of ρ and R_H (the latter used to derive n_o , both required to get the Hall mobility), we have set and attained two other goals: (1) to make such measurements over a significant temperature range, and (2) to correct near-intrinsic samples for ambipolar (hole) conduction, so that n_o itself and μ_{Hn} itself are obtained.

A procedure developed by this writer and his former student (Tang et al. 1989) describes how an ambipolar correction can be made when the ratio (R_{Hi}/R_H) of intrinsic to actual Hall coefficient is not much larger than unity; i.e., when the sample is near-intrinsic. For many of the VZM-grown samples we measured during the course of this NRL program, the correction was numerically very small, but in some cases it did make a difference. In any event, our computer program can do this at the touch of a key, so we do it routinely. This program has the additional advantage that it evaluates the fraction of midgap donor EL2 centers which are ionized: $P_i = (N^+/(N^+ + N^o)) = (N^+/N_{EL2})$.

Measurement of the dc electrical properties versus temperature provides the best assurance as to whether or not the properties of an undoped GaAs sample are truly controlled by the partial compensation of EL2. For if they are, the value of P_i will not vary much upon warming, maybe decrease to a small extent. On the other hand, if P_i is too small (not enough compensating acceptors), the Fermi energy "floats" too high to be under EL2 control, the activation energy of $n(T)$ is smaller than EL2 would dictate, and the observed value for P_i rises with temperature.

Figure 1 (on page 5) shows data of $\log(n_o)$ versus $(1000/T)$ for samples from Sections II and III of VZM Crystals 26-41N and 26-52N; these samples were measured in the spring of 1991, and first reported to NRL at that time in detail. It can be seen from the figure that (1) The slope (activation energy) is about the same for all four samples, the slope indicative of full control by EL2. Also, (2) especially for samples 26-41N-II-8 and 26-52N-II-3, the ratio (n_o/n_i) is less than three. For those near-intrinsic samples, the ambipolar correction did amount to a finite percentage. The larger P_i is (i.e., the more numerous the shallow acceptors such as C_{As}), the more the line for $n_o(T)$ is pulled down towards the intrinsic line; and we have in the past seen examples of "undoped" SI GaAs for which the value of (n_o/n_i) was slightly more than 1.0 at room temperature, declining to slightly less than 1.0 on warming! All this is caused by the EL2 defect control.

Such a large value of P_i (the case just described requires $P_i \approx 0.5$) is neither necessary nor desirable, for one has not only a 300 K resistivity of some 10^9 ohm cm, but is apt to have carrier mobility degraded by the substantial population of ionized acceptors, which scatter carriers even more efficiently than ionized EL2 traps. Yet a value of P_i which is too small (say, less than 0.05) is also non-ideal, since small variations of P_i in space then degrade the mobility because of the little potential barriers they create for electrons. Those variations in P_i arise only to a small extent because of spatial variations of N_{EL2} itself, they are much more the result of $(N_a - N_d)$ of shallow impurities varying on a micron distance scale.

[Text resumes on page 6]

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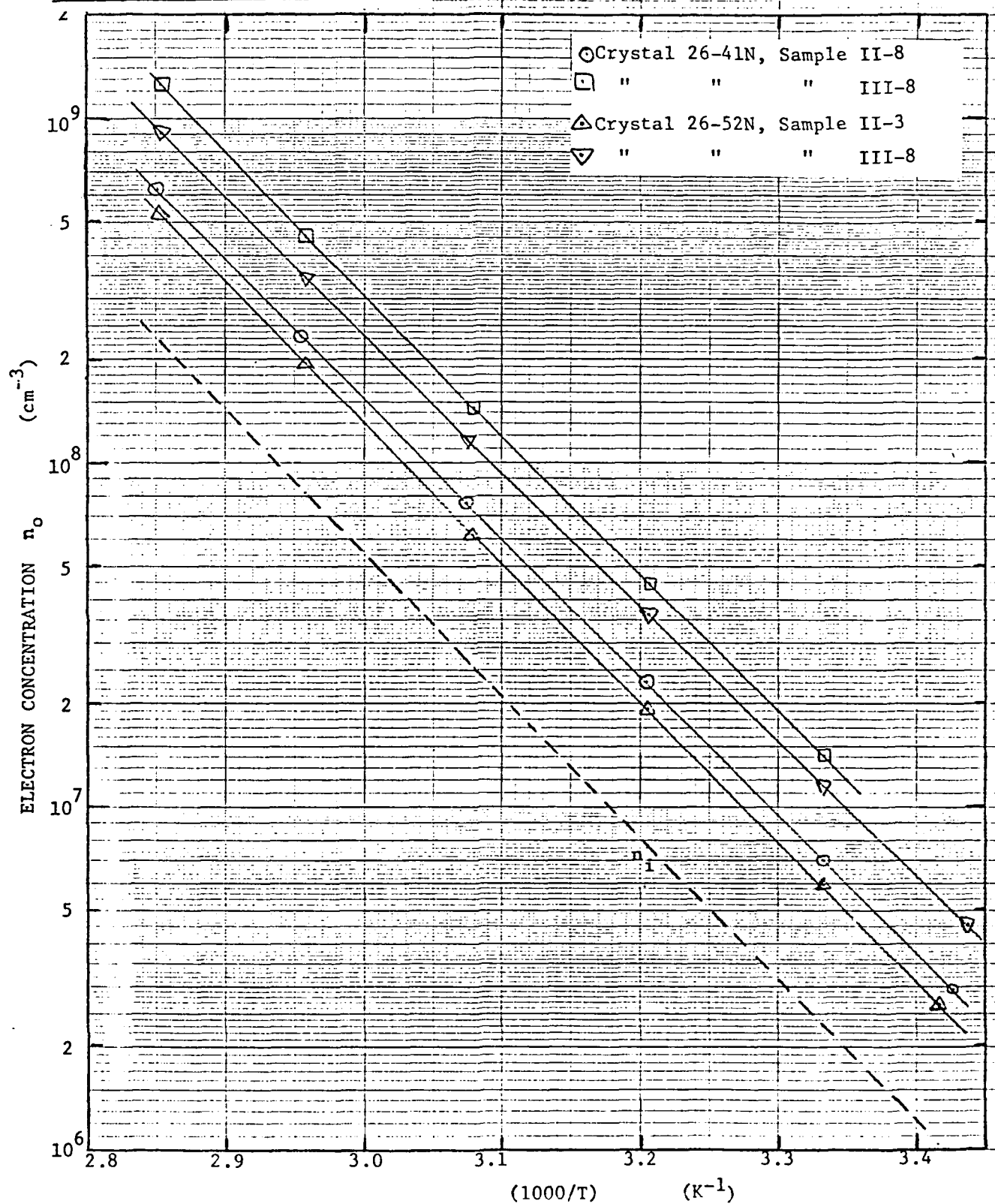


Figure 1. Electron concentration (ambipolar corrected) vs. reciprocal temperature for the four samples from Crystals 26-41N and 26-52N.

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Thus in the charge compensation balance equation for SI GaAs:

$$N^+ = P_i N_{EL2} = (N_a - N_d) + (n_o - p_o) \quad (1)$$

one expects, of course, that the material is trap-dominated to the extent that the free carrier term $(n_o - p_o)$ is trivially small compared with the other quantities, but one also wants the quantity $(N_a - N_d)$ to be consistently and spatially-independently positive. That desire^a is less easy to fulfil as one makes N_d and N_a smaller, so that P_i [and hence the energy $(\epsilon_c - \epsilon_v)$] tends to fluctuate. The^a lack of EL2 compensation control shows up when dc transport measurements are made versus temperature, (i) in $n(T)$ having an activation energy smaller than the 0.77 eV value characteristic of EL2, and (ii) in the mobility being sub-normal, perhaps with μ_{Hn} creeping slightly larger as T rises. (That happens since the obstructive efficiency of small potential barriers is lessened as kT rises.) Those who measure only at one temperature near to 300 K can only guess at these things.

Accordingly, we have reported variable-temperature transport data for samples from all of the NRL-grown crystals studied in the program, including the data for 4-88H and 27-19N to be found later in this Report. Transport data do not yield a number for the concentration of EL2 defects (this we get from near-IR optical data as noted below), but do yield the value for P_i , needed in addition to optical data for evaluation of $N^+ = P_i N_{EL2}$.

Near-IR Transmittance for EL2 Concentration

This is a measurement the writer has been involved with for the past eight years. For a "clean" sample of "undoped" (i.e., EL2 controlled) SI GaAs, prepared as a parallel-faced polished slab, the spectral dependence of the optical absorption coefficient in the near-IR just below bandgap ($0.8 < h\nu < 1.3$ eV) is dominated by the absorption of neutral and ionized EL2 centers

$$\left. \begin{aligned} \alpha(h\nu) &= [N^o \sigma_I^o(h\nu) + N^+ \sigma_N^o(h\nu)] \\ &= N_{EL2} [(1 - P_i) \sigma_I^o(h\nu) + P_i \sigma_N^o(h\nu)] \end{aligned} \right\} \quad (2)$$

where $\sigma_I^o(h\nu)$ is the photo-ionization cross-section for any neutral EL2 center, and $\sigma_N^o(h\nu)$ is the photo-neutralization cross-section for a center already ionized. The first of those cross-sections was determined in magnitude and spectral form by Martin (1981). The spectral form of σ_N^o has been known from photocapacitance studies more than a decade ago, but the actual scaling for this relatively weak absorption mechanism was not pinned down until much more recently (Silverberg et al., 1988; Blakemore et al., 1989).

At any rate, if absorption connected with other trap species and any other optical processes is small enough to be neglected, then one can use data of $\alpha(h\nu)$ to determine N^o (at least), and maybe deduce both N^o and N^+ . The latter can be done most efficiently if one already has knowledge of P_i from electrical data.

Our measurements are made of the transmitted fraction $T \equiv (I_t/I_i)$ of light as wavelength is scanned. There are large reflection losses, since GaAs has a reflectivity $R \approx 0.3$ at both front and rear surfaces, and multiple reflections occur. Provided that the instrumental resolution employed is not so high as to

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make the optical coherence length larger than the sample thickness 't', then the various transmitted components add up by intensities, resulting in

$$T = (I_t/I_o) = (1 - R)^2 \exp(-\alpha t) [1 - R^2 \exp(-2\alpha t)]^{-1} \quad (3)$$

This is actually a quadratic equation for $\exp(\alpha t)$, and so from transmittance data (knowing the reflectivity vs. wavelength), one can calculate $\alpha(\lambda)$ or $\alpha(h\nu)$, which we then plot out. Our system acquires data every 0.5 nm wavelength step, usually from 1.0 to 1.45 μm , resulting in a 900-point plot of $\alpha(\lambda)$ for that range; or equivalently from 855 meV to 1.24 eV, for $\alpha(h\nu)$.

Figure 2, on page 8, shows such a plot, for the optical sample from Section IV of Crystal 26-11N. This was a crystal grown with a deliberate excess of As in the ampoule, to see if a Ga \rightarrow As shift in the stoichiometry would tend to produce a larger EL2 concentration. In this case, it certainly worked, since the resulting N_{EL2} (about $1.6 \times 10^{16} \text{ cm}^{-3}$) is about twice as large as usually happens with the VZM-grown GaAs crystals. Most of the crystals listed in Table I were grown with one or more of the growth parameters adjusted in order to find out what happens to the material grown under that changed condition.

For the sample whose near-IR absorption appears in Figure 2, the amount of absorption to be associated with EL2^+ photo-neutralization was not large enough to show up appreciably. That phenomenon is supposed to have a fairly broad peak centered on 0.95 eV. Its effect on $\alpha(h\nu)$ is prominent only for material in which P_i is rather large. An example is shown in Figure 3, on page 9. This shows the absorption for a sample from near the lower end (Section II) of Crystal 27-1N. This sample also showed an appreciable presence of carbon from mid-IR absorption (see below), and of course C_{As} acceptors would have a major influence on $(N_a - N_d)$ and P_i in Eq.(2). Just to show how chancy this evaluation business is, I must comment that the value (~ 0.2) for P_i one would infer from the scalings in Figure 3 is appreciably smaller than the number for P_i we later got from analysis of a nearby electrical sample. Nevertheless, we try and persist. The more types of data one has to compare, the better the chance of getting a realistic appraisal.

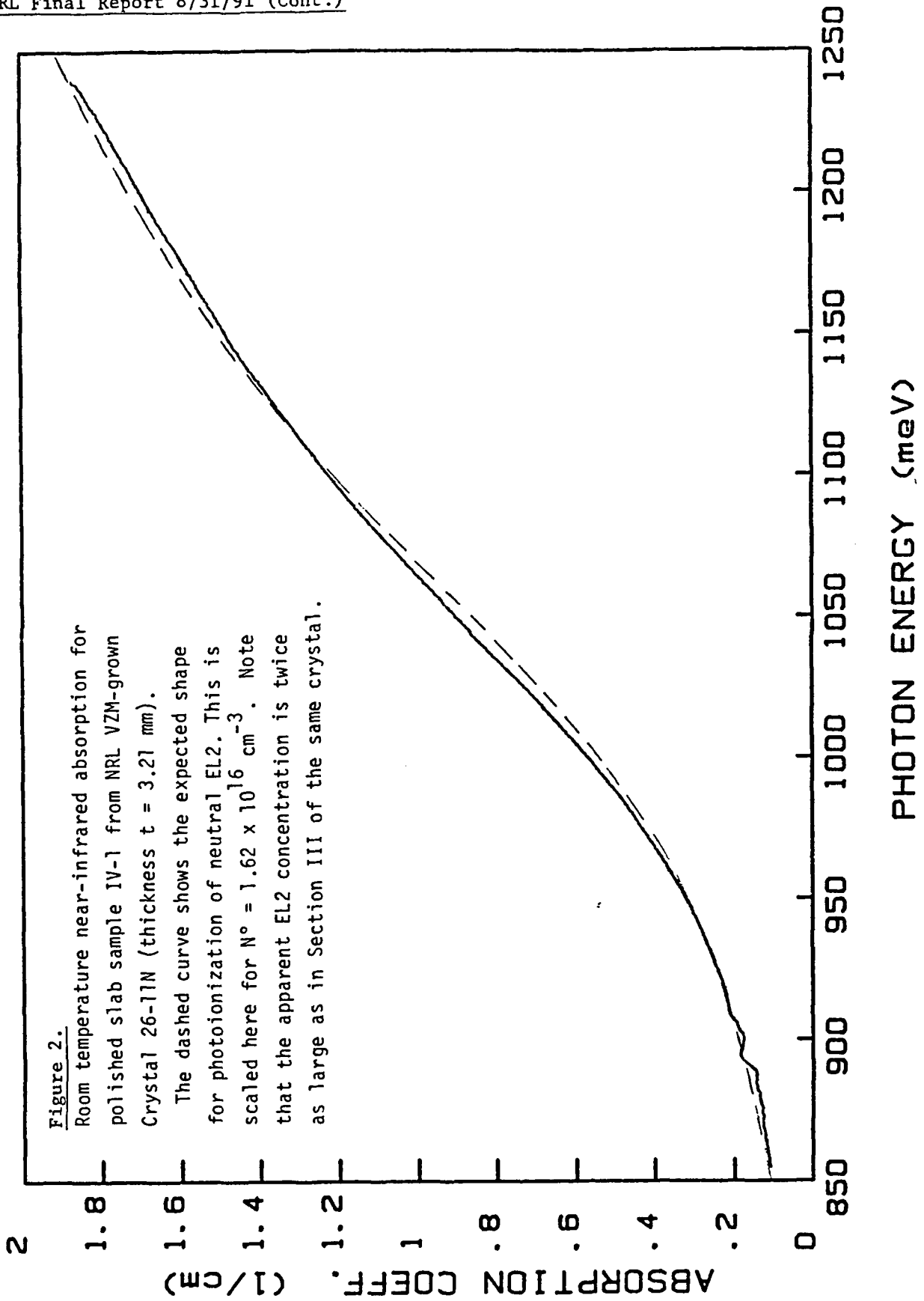
Mid-IR Observations for Carbon LVM Absorption

Almost any impurity or defect in a crystalline solid should have some special vibrational mode (LVM) with some potentiality for this to be optically excited. The efficiency varies widely, but is always pretty weak! A complication is that the spectral locations of LVM bands tend to overlap the multiphonon lattice absorption spectrum. This is true for C_{As} acceptors in GaAs ($\bar{\nu} = 580 \text{ cm}^{-1}$ at room temperature), and the situation is far more dubious again for Si_{Ga} donors in GaAs. Thus for all practical purposes, our mid-IR work is limited to attempts to detect carbon in its predominant C_{As} acceptor form.

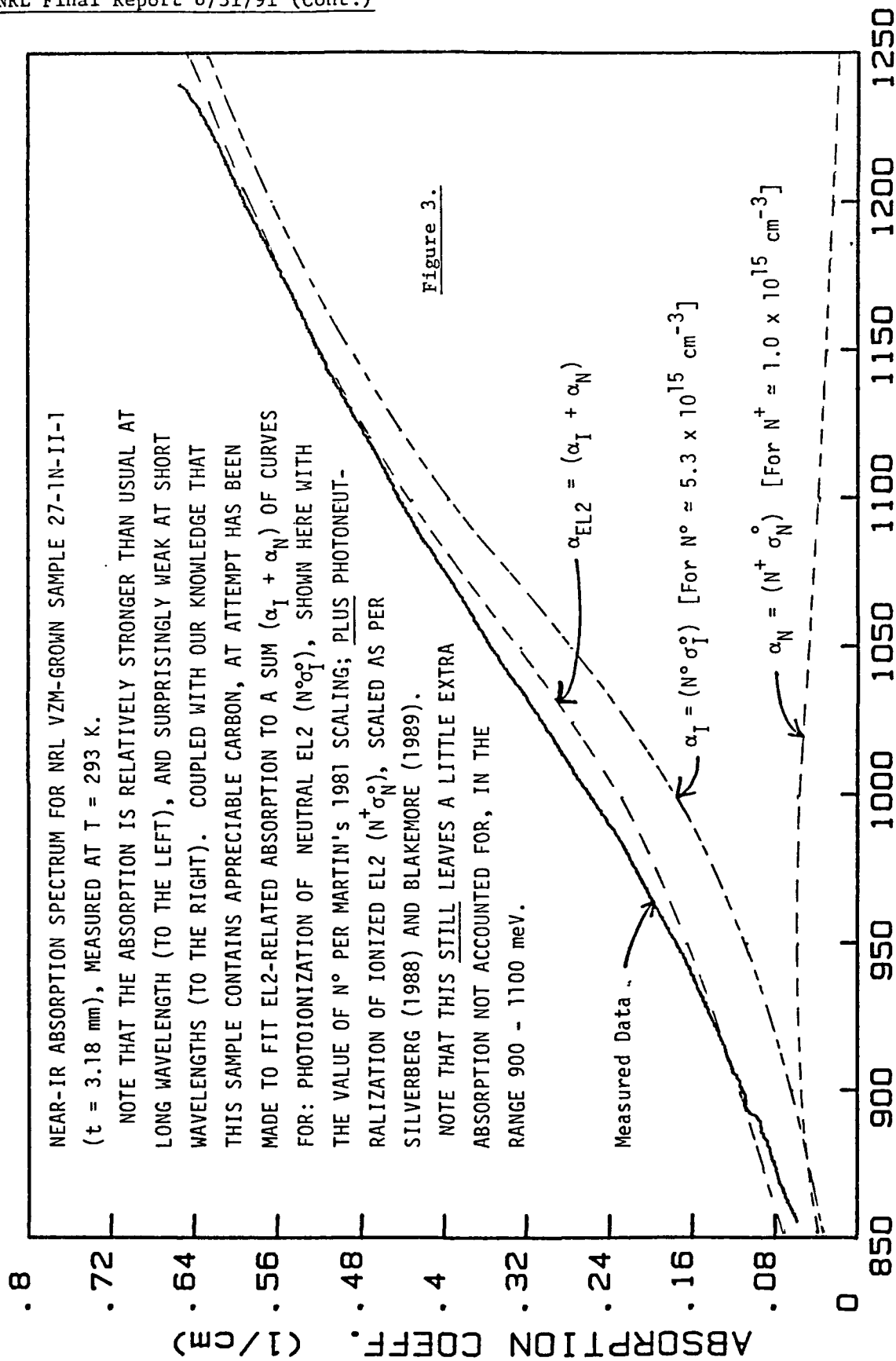
The raw data taken in such an observation consists of a plot of $T = (I_t/I_o)$ versus wavenumber for a spectral range including the desired LVM band. In carbon observation, we make a plot from 600 to 550 cm^{-1} (see Figure 4 on page 10), or just from 590 to 570 cm^{-1} (see Figure 5 on page 11) to study the immediate LVM region at larger magnification. There is an overall trend for T to decrease as $\bar{\nu}$ decreases, due to increased strength of lattice absorption; thus one looks for an extra dip of transmittance centered on about 580 cm^{-1} wide. This dip can be seen clearly in Sample II-1 of Crystal 27-1N, in Figures 4 and 5; note that this

[Text resumes on page 12]

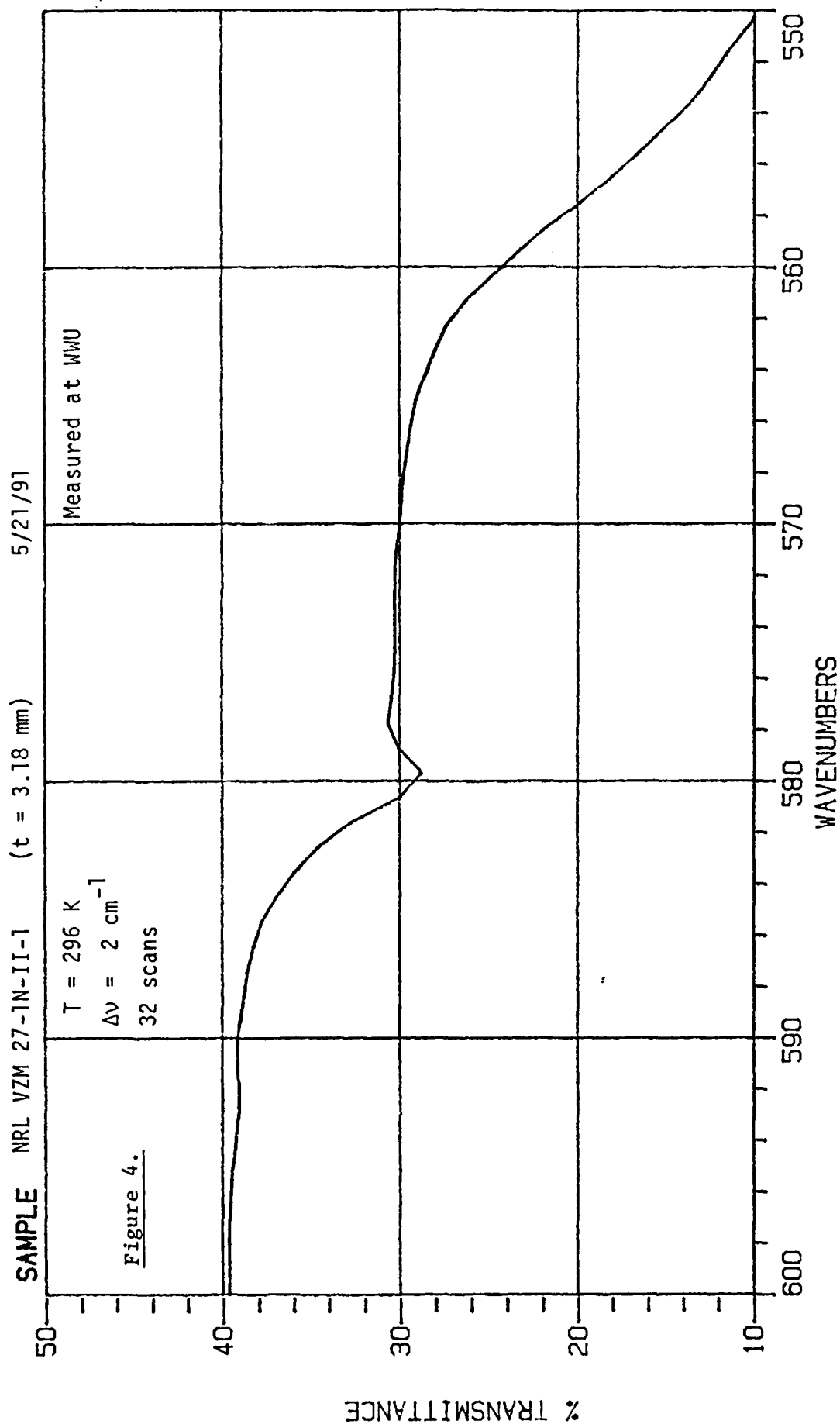
SAMPLE : N26-11N-IV-1 DATE : 08/30/90

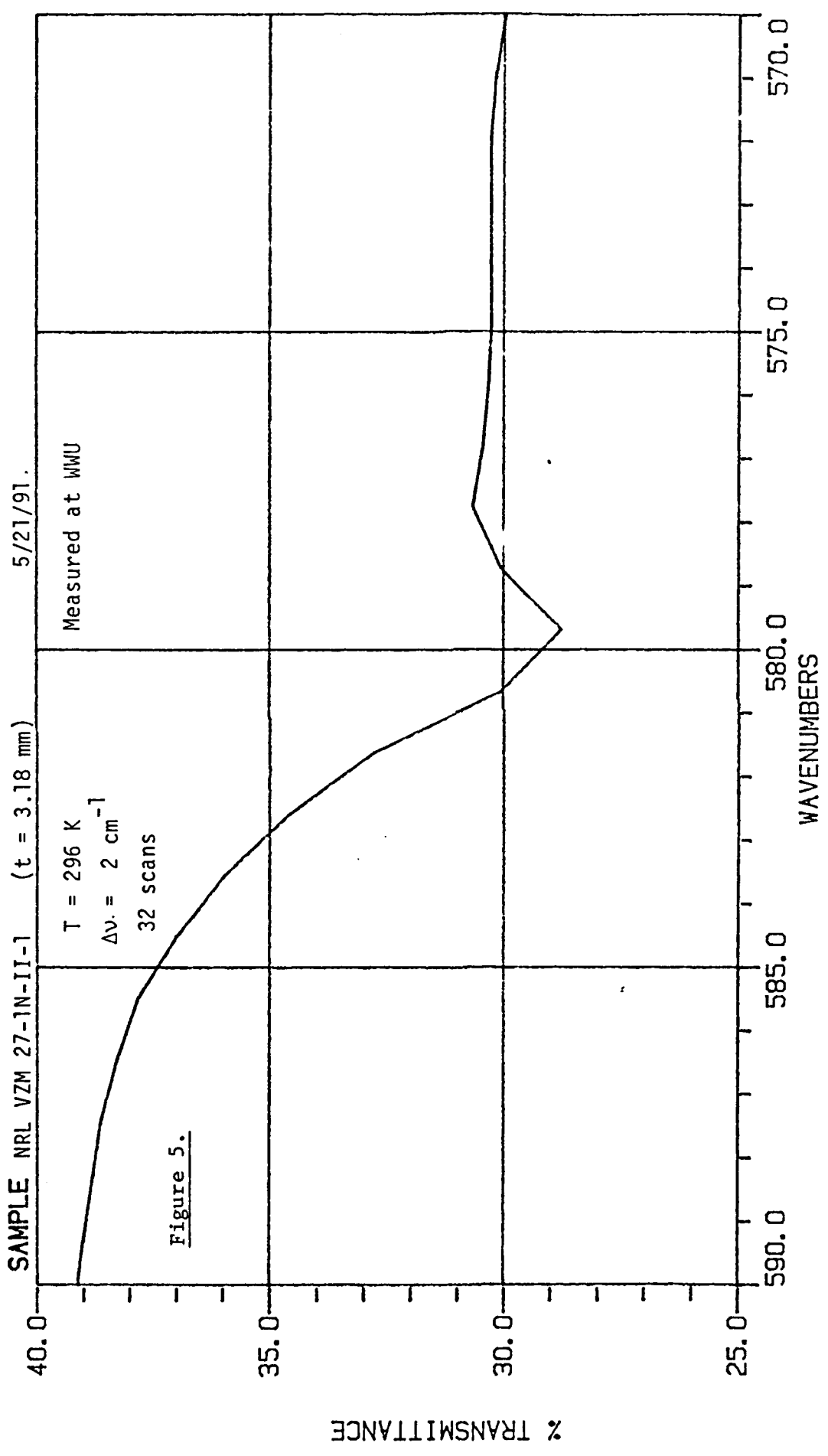


SAMPLE : VZM-27-1N-II-1 DATE : 05/23/91



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is the same polished slab for which the near-IR data of Figure 3 show a substantial fraction of the EL2 to be ionized (and for which electrical data indicated an ever larger P_i). Thus Sample 27-1N-II-1 is just the type of SI GaAs sample for which a detectable amount of carbon LVM absorption might be expected.

The data of Figures 4 and 5 were obtained, as is usual these days, with a FTIR instrument. The FTIR which has been available to us at WWU up until now is a bottom-of-the-line Nicolet, with only a 2 cm^{-1} resolution. Thus the 580 cm^{-1} transmittance "dips" in these figures are broadened compared with the natural LVM bandwidth, and one could not transform those data to get meaningful numbers for N_{Carb} . That situation is in the process of change, since WWU is now purchasing a new Mattson FTIR with 0.5 cm^{-1} resolution and wideband MCT detector. The transmittance curves obtainable with that new instrument, which should be up and running in the Fall of 1991, can then be computer-converted into curves of absorption coefficient versus wavenumber, and a numerical integration made of the LVM band area with respect to the lattice absorption "baseline" curve. From the band area, one can then extract a number for N_{Carb} (Sargent and Blake-more 1989), even with room temperature measurements.

Measurements at 0.5 cm^{-1} resolution were made during the Fall of 1990 with the optical samples from three of the VZM crystals studied in this program: Crystals 26-5N, 26-11N, and 26-16N. The type of numerical integration noted above was done for these laboriously by hand, since the necessary raw data had been obtained using the Bomem FTIR at Simon Fraser University in Canada, and I was not equipped to do computer analysis on that data in its core memory form.

For the other ten crystals listed in Table I, the measurements were done at WWU with the 2 cm^{-1} resolution, and left simply as traces of transmittance, from which one can infer carbon presence to be "strong" (as with 27-11N-II-1), "moderate", "weak", or "very weak to undetectable". This assessment is still useful as a cross-check on the results obtained using electrical and near-IR data. However, it is hoped that in a follow-on program, we shall be able to provide more quantitative carbon information, thus supplementing the low-temperature measurements Dr. W. J. Moore makes at NRL.

Having now described the various types of measurement used in this NRL-funded program, I now go on to report on the two most recently-received sets of samples.

Report on Samples from Crystal 4-88H.

Two optical and two electrical sample wafers were received from this VZM crystal on August 7 1991. While grown in a VZM furnace at NRL, as with many other samples noted in Table I, these samples are of interest in having come from a GaAs crystal grown in Dr. Richard Henry's furnace rather than in Dr. Nordquist's "original GaAs" VZM furnace. A 1-page table (repeated here as Table II on page 13) summarizes the room temperature properties found for these samples, as already sent to NRL on August 16. The evaluation of those samples is now provided here in fuller tabular and graphical detail.

The first measurements made by this writer and my student assistant Mr. Steven Saban for this crystal were of the mid-IR range by FTIR; as usual at 2 cm^{-1} resolution. Figures 6 and 7 (on pages 14 and 15) show the course of the Sect. II transmittance for the $600 - 500$ and $590 - 570\text{ cm}^{-1}$ ranges, and these show that evidence for carbon LVM absorption is present, but weak. Figures 8 and 9 (on pages 16 and 17) show the corresponding traces for the optical sample from Section III of Crystal 4-88H, with any carbon absorption vanishingly weak.

[Text resumes on page 18]

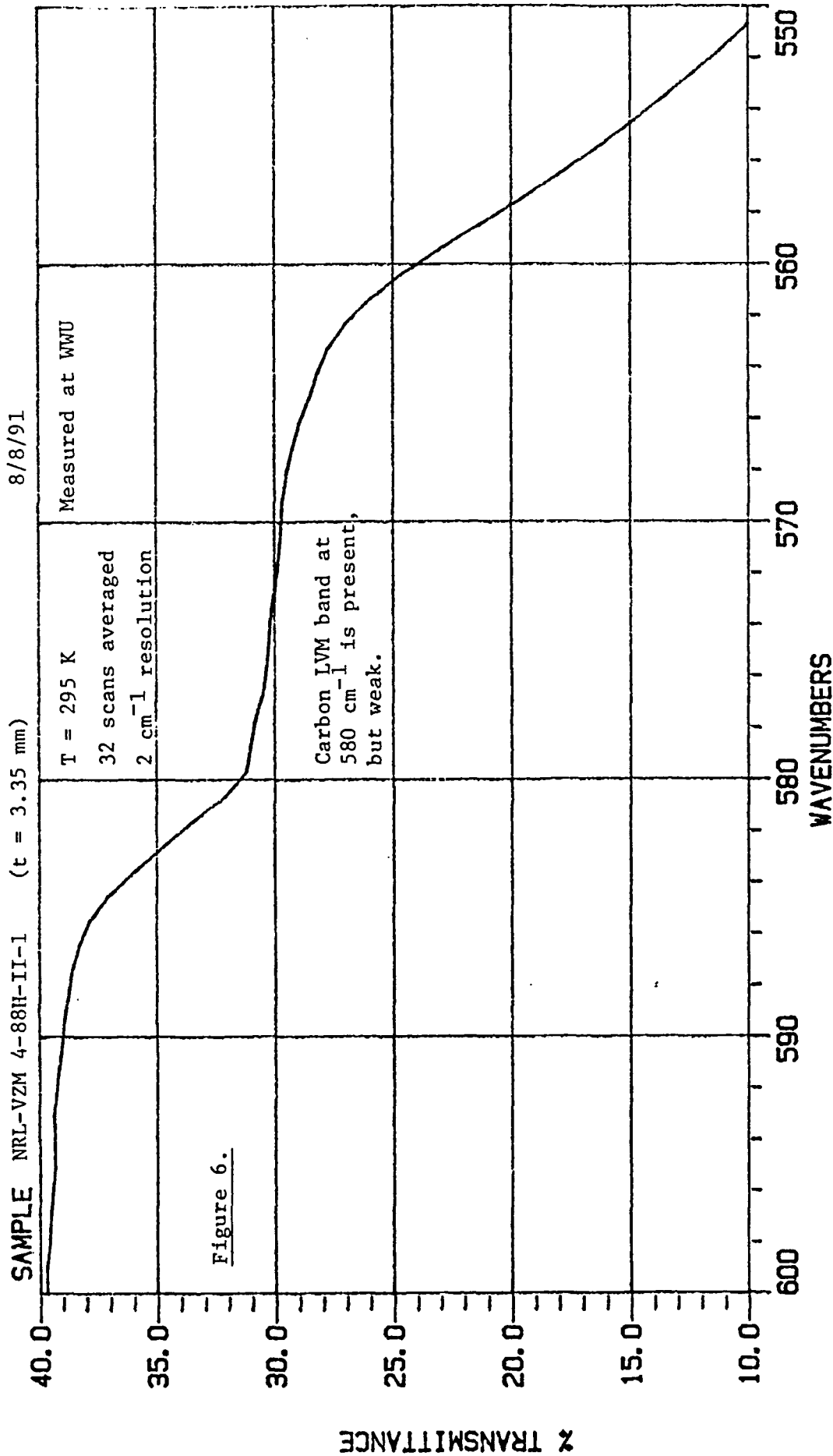
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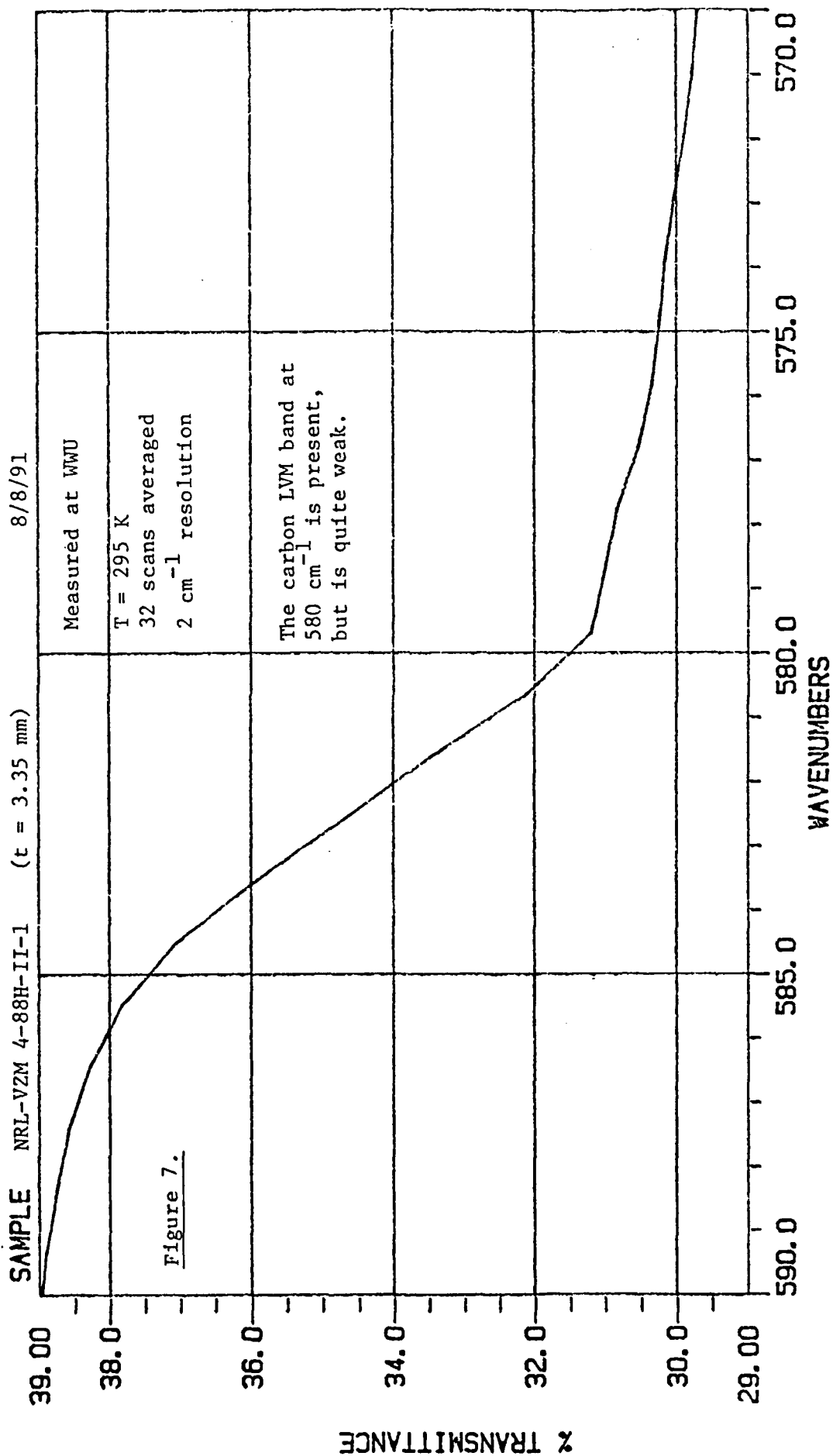
Table II. Summary of Room-Temperature Properties for Samples from Undoped VZM-Grown Crystal 4-88H. Data Obtained August 1991.**

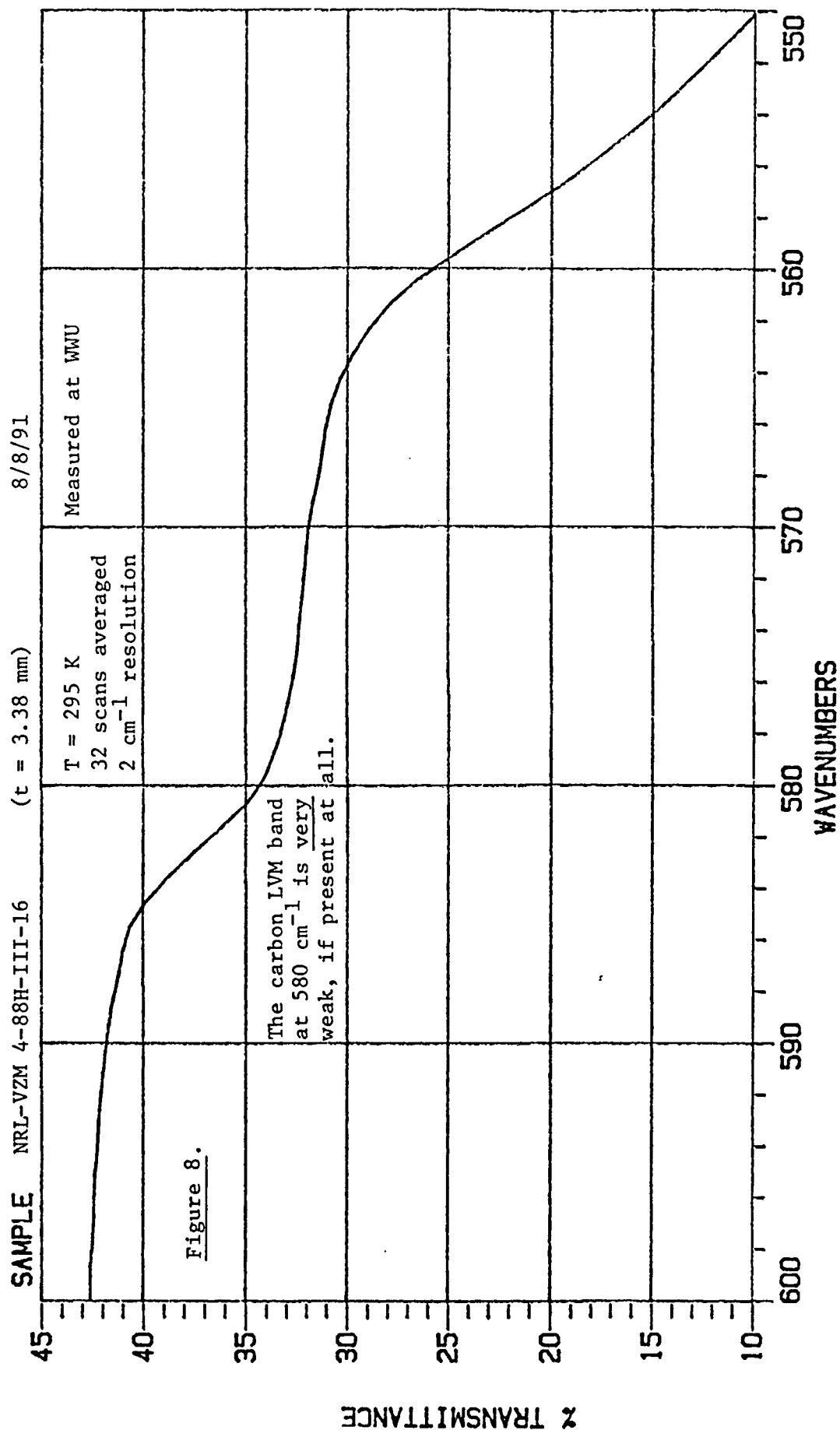
[Note: This crystal was grown in Dr. Richard Henry's VZM growth system, and thus provides an opportunity for comparison with numerous other undoped GaAs crystals VZM-grown in Dr. Paul Nordquist's furnace.]

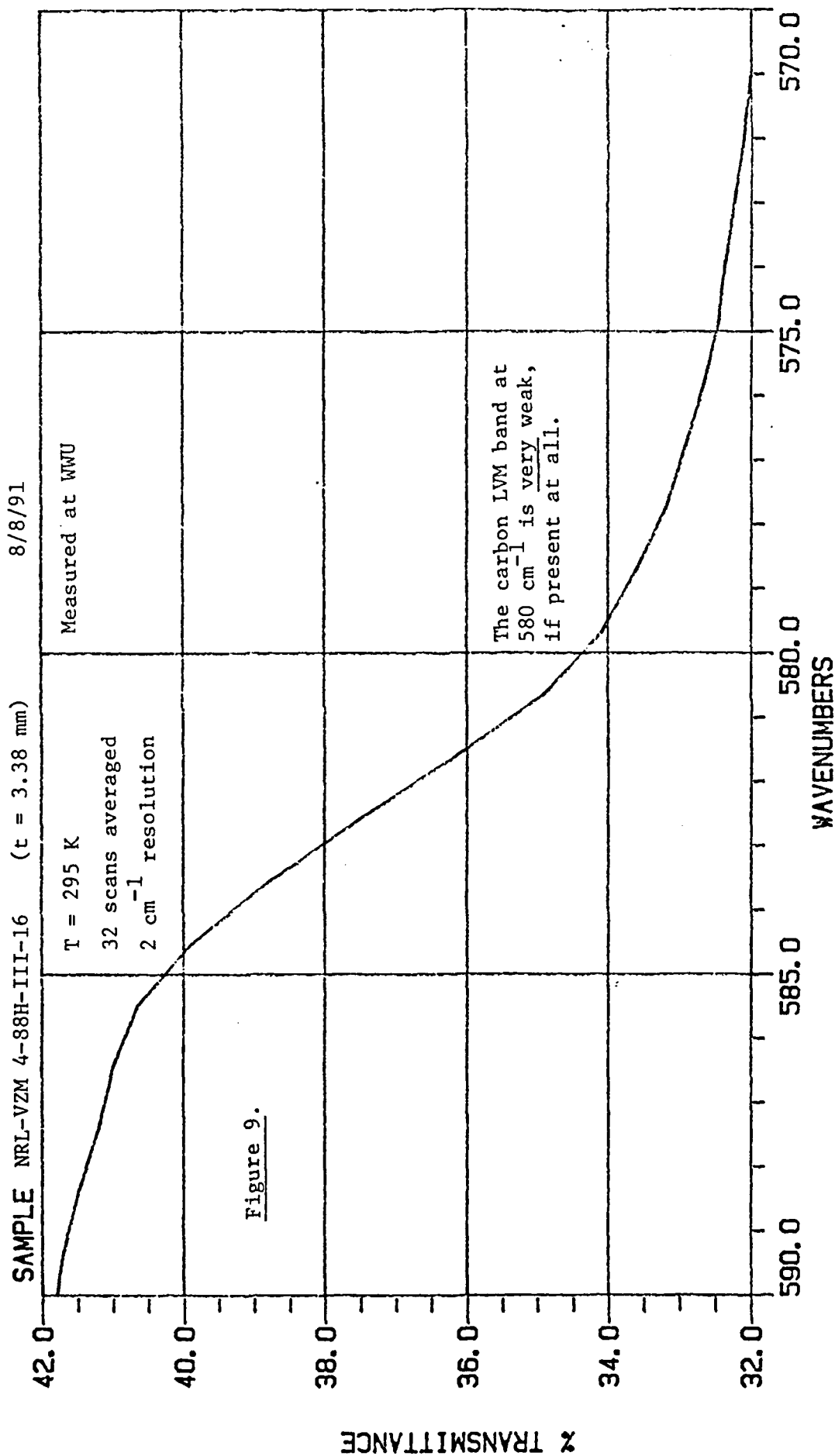
Crystal	VZM 4-88H	
Optical Sample Slab	II-1	III-16
Electrical Data Wafer	II-7	III-9
300.0K Electrical Data:-		
Resistivity ρ (ohm cm)	2.10×10^7	9.62×10^6
Hall Coeff. R_H ($\text{cm}^3/\text{Coulomb}$)	-9.23×10^{10}	-3.09×10^{10}
Ratio $z = (R_{Hi}/R_H)$	31.3	94
Ambipolar-corrected Electron Hall Mobility μ_{Hn} ($\text{cm}^2/\text{V s}$)	4388	3209
Ambipolar-corrected Electron Concentration n_o (cm^{-3})	7.98×10^7	2.39×10^8
Ratio (n_o/n_i)	34.7	104
EL2 Ionized Fraction P_i	0.0377	0.0108
EL2 Concentrations (Neutral, Ionized, Total) Deduced from the above P_i values, combined with room temp (~ 295 K) near-IR absorption data:		
1.1 μm Absorption Coeff. $[\alpha (\text{cm}^{-1})]$	0.645	0.640
Deduced Neutral N^o (cm^{-3})	7.5×10^{15}	7.5×10^{15}
" Ionized N^+ (cm^{-3})	3×10^{14}	1×10^{14}
Thus, Total EL2 Concentration $N_{\text{EL2}} = (N^o + N^+) (\text{cm}^{-3})$	7.8×10^{15}	7.6×10^{15}
Evidence of Carbon (C_{As}) from 580 cm^{-1} room temperature FTIR observations (LVM band)	Present, but weak	Very weak, if present at all.

** Please see other tables and figures for electrical properties versus temperature for this crystal, plus the figures with optical data for Samples II-1 and III-16, used above.









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Since the carbon concentration was (at best) quite small in this crystal, it could be expected that the near-IR spectral dependence of optical absorption would be a good fit just to the neutral EL2 photoionization term in Eq.(2). This proved to be the case. Figure 10 (on page 19) shows the variation of $\alpha(h\nu)$ for Sample II-1, while Figure 11 (on page 20) shows the corresponding curve for Sample III-16. Note that each curve fits quite well to the form described by Martin (1981) for EL2 photoionization alone. (The artifact around 900 meV in Figure 10, present but much weaker in Figure 11, is caused by water vapor absorption in the laboratory, and is nothing to do with the GaAs sample.)

I was not advised by Dr. Henry that in the growth of this crystal anything was done to perturb the "ordinary" stoichiometry of Ga-As, thus one would expect the N_{EL2} to lie in the "normal" range for a VZM crystal for both Sections II and III. This was indeed the case. The curve-fitting in Figures 10 and 11 indicates $N^o \approx 7.6$ to $7.7 \times 10^{15} \text{ cm}^{-3}$ for the two samples. The actual numbers for absorption coefficient at 1127 meV ($1.1 \mu\text{m}$) are noted in the respective columns in Table II, and Eq.(2) for that spectral location are used (in conjunction with electrically determined P_i) to deduce numbers for N^o , N^+ , and their sum, N_{EL2} . As worked out by that method, one ends up with a total EL2 concentration of 7.8×10^{15} for Sample 4-88H-II (approximately 4% ionized), and one of $7.6 \times 10^{15} \text{ cm}^{-3}$ for Sample 4-88H-III (only 1% ionized). As the electrical results reported below demonstrate, the material of Section III from Crystal 4-88H did not have enough acceptors to keep the Fermi energy controlled by EL2.

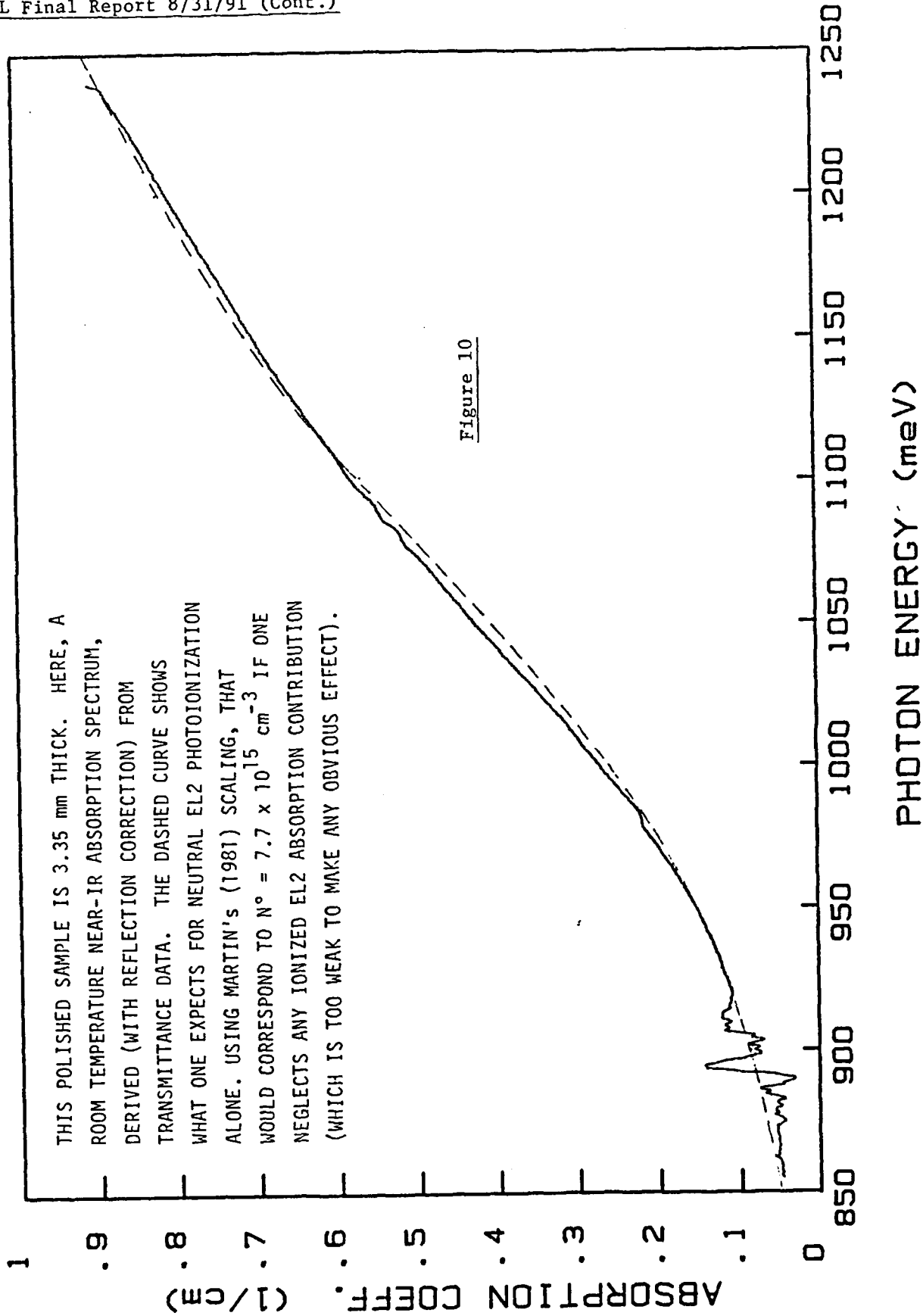
Sample 4-88H-II-7 was measured at 300.0 K and at five other temperatures, with results as tabulated in Table III, on page 21. For this sample, the ratio $P_i = (N^+/N_{EL2})$ was rather small (less than 0.04), but the EL2 defect donor level was in control. A few more carbon acceptors might have made the situation of this sample less precarious, but there were enough for this to be convincingly SI in behavior, with the influences of minor defect species other than EL2 held in abeyance. As will later in this report be shown graphically (in Figure 19), the mobility for Sample 4-88H-II-7, though not as high as some GaAs is capable of, does show the downwards trend of μ_{Hn} with rising T which is the ordinary evidence that microscopic potential barriers - while probably present - are not in full control of electron transport.

Things were different for Sample 4-88H-III-9, as shown in tabular form in Table IV on page 22. As compared with Sample II-7 from the same crystal, the 300 K electron concentration is only three times larger, but the essence of EL2 compensation control has been lost. Table IV shows that the value of P_i moves upwards from ~1% at 300 K to 2% by 350 K. The graphical evidence of n_o versus reciprocal temperature (shown later, in Figure 20) shows that this sample has an activation energy significantly smaller than that EL2 would produce. In short, the Fermi level is not under EL2 control, and has risen to become ensnared by a minor defect level: note that ϵ_F with respect to midgap hovers around 160 meV over that 50°C temperature range. The presence of potential barriers as the features limiting and controlling the mobility is also manifest from the quite low 300 K mobility, and its gradual climb upwards as temperature rises. (See Table IV and Figure 19.)

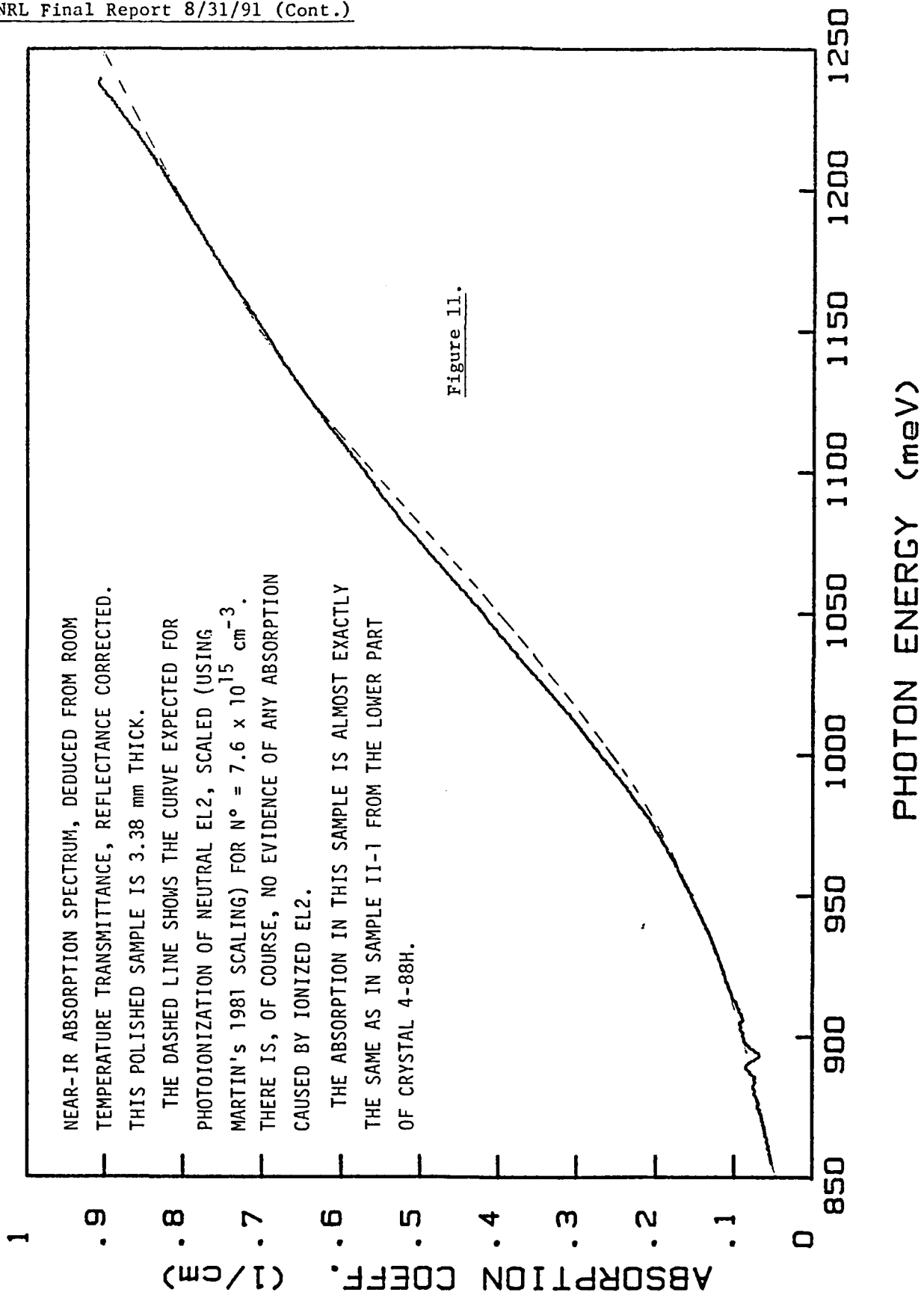
And so Crystal 4-88H is an example of a GaAs crystal whose SI character would be improved by having (a few) more shallow acceptors present. Rather than just adding carbon, one might do better to include a tiny amount of some acceptor dopant whose solid:liquid segregation coefficient was less than unity, such as zinc. The amount required would need to be only enough to provide a Zn_{Ga}

[Text resumes on page 23]

SAMPLE : V4-88H-II-1 DATE : 08/09/91



SAMPLE : V4-88H-III-16 DATE : 08/12/91



NRL Final Report 8/31/91 (Cont.)

Table III Electrical data vs. Temperature for Sample 4-88H-II-7 ($t = 0.58$ mm)
From Undoped VZM-Grown GaAs Crystal 4-88H. (Data of 8/13/91).

[Note: This crystal was grown in Dr. Henry's furnace system. The first set of columns shows the as-measured "raw" electrical data, plus conversion from R_H into n_o using a T-dependent r_{Hn} but no ambipolar correction. The second set of columns shows the effect (numerically negligible in this sample) of making an ambipolar correction. The third set of columns carries this through to make a numerical evaluation of the EL2 ionized fraction. Here, $P_i \approx 3\%$.]

TEMP. (K)	RESISTIVITY (OHM-CM)	HALL COEFFICIENT (CM ³ /COUL.)	HALL MOBILITY (CM ² /VS)	CARRIER CONCENTRATION (CM ⁻³)	1000/T (1/K)
295.1	3.434E+7	-1.515E+11	4412	4.877E+7	3.388
300	2.103E+7	-9.228E+10	4388	7.981E+7	3.333
312.2	6.750E+6	-2.929E+10	4338	2.494E+8	3.203
324.8	2.278E+6	-9.776E+9	4292	7.407E+8	3.078
337.9	7.765E+5	-3.243E+9	4176	2.212E+9	2.959
350.2	3.110E+5	-1.283E+9	4124	5.546E+9	2.855

TEMP. (K)	RESISTIVITY (OHM-CM)	HALL COEFFICIENT (CM ³ /COUL.)	HALL MOBILITY (CM ² /VS)	CARRIER CONCENTRATION (CM ⁻³)	1000/T (1/K)	Z
295.1	3.434E+7	-1.515E+11	4412	4.876E+7	3.388	32.41
300	2.103E+7	-9.228E+10	4388	7.980E+7	3.333	31.34
312.2	6.750E+6	-2.929E+10	4339	2.493E+8	3.203	28.3
324.8	2.278E+6	-9.776E+9	4292	7.406E+8	3.078	25.63
337.9	7.765E+5	-3.243E+9	4176	2.212E+9	2.959	24.38
350.2	3.110E+5	-1.283E+9	4125	5.545E+9	2.855	22.48

SAMPLE NAME = V4-88H-II-7

DATE = 08/13/91

AMBIPOLAR CORRECTION DATA

T (K)	Ei (mev)	Phi (mev)	Ni (cm ⁻³)	No (cm ⁻³)	Nd* (cm ⁻³)	Ed (mev)	Ef (mev)	Pi 1/(1+No/Nd*)
295.1	712.3	40.3	1.362E+6	4.876E+7	1.574E+6	43.9	131.2	.0313
300	711.2	40.9	2.303E+6	7.980E+7	2.608E+6	44.1	132.6	.0317
312.2	708.5	42.6	7.941E+6	2.493E+8	8.578E+6	44.7	135.3	.0333
324.8	705.6	44.3	2.595E+7	7.406E+8	2.677E+7	45.2	138.1	.0349
337.9	702.6	46.1	8.119E+7	2.212E+9	8.005E+7	45.7	142.3	.0349
350.2	699.7	47.8	2.198E+8	5.545E+9	2.082E+8	46.1	145.2	.0362

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Table IV. Electrical Data vs. Temperature for Sample 4-88H-III-9 ($t = 0.62$ mm)
From Undoped VZM-Grown GaAs Crystal 4-88H. (Data of 8/14/91).

[Note: This crystal was grown by Dr. Henry in his VZM furnace. The first set of columns below shows as-measured raw electrical data, plus conversion from R_H to n_o using a T-dependent r_{Hn} . The second set of columns shows n_o and mobility as ambipolar-corrected; of no significance at all for this far-from-intrinsic sample. The third set of columns includes evaluation of EL2 ionized fraction P_i . Due to the smallness of N_a , EL2 control is not firmly held, exemplified by the 2:1 decrease of $z = (R_{Hi}/R_H)^a$, and increase of P_i , with rising T.]

TEMP. (K)	RESISTIVITY (OHM-CM)	HALL COEFFICIENT (CM ³ /COUL.)	HALL MOBILITY (CM ² /VS)	CARRIER CONCENTRATION (CM ⁻³)	1000/T (1/K)
300	9.621E+6	-3.088E+10	3209	2.385E+8	3.333
312	3.420E+6	-1.177E+10	3441	6.207E+8	3.205
325.1	1.171E+6	-4.262E+9	3637	1.699E+9	3.075
338	4.379E+5	-1.654E+9	3778	4.337E+9	2.958
350	1.849E+5	-7.130E+8	3857	9.979E+9	2.857

TEMP. (K)	RESISTIVITY (OHM-CM)	HALL COEFFICIENT (CM ³ /COUL.)	HALL MOBILITY (CM ² /VS)	CARRIER CONCENTRATION (CM ⁻³)	1000/T (1/K)	Z
300	9.621E+6	-3.088E+10	3209	2.385E+8	3.333	93.66
312	3.420E+6	-1.177E+10	3441	6.207E+8	3.205	71.85
325.1	1.171E+6	-4.262E+9	3637	1.699E+9	3.075	57.22
338	4.379E+5	-1.654E+9	3778	4.337E+9	2.958	47.38
350	1.849E+5	-7.130E+8	3857	9.978E+9	2.857	41.08

SAMPLE NAME = V4-88H-III-9

DATE = 08/14/91

AMBIPOLAR CORRECTION DATA

T (K)	Ei (mev)	Phi (mev)	Ni (cm ⁻³)	No (cm ⁻³)	Nd* (cm ⁻³)	Ed (mev)	Ef (mev)	Pi 1/(1+No/Nd*)
300	711.2	40.9	2.303E+6	2.385E+8	2.608E+6	44.1	160.9	.0108
312	708.5	42.6	7.787E+6	6.207E+8	8.419E+6	44.7	160.3	.0134
325.1	705.5	44.4	2.666E+7	1.699E+9	2.748E+7	45.2	160.7	.0159
338	702.5	46.1	8.187E+7	4.337E+9	8.070E+7	45.7	161.7	.0183
350	699.7	47.7	2.164E+8	9.978E+9	2.051E+8	46.1	163.3	.0201

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of (say) $\sim 5 \times 10^{14} \text{ cm}^{-3}$, to ensure an EL2 compensation approaching but not having to exceed 10%. I should note in passing that trace zinc doping was used by Cominco (and their follow-on name Johnson Matthey) as an acceptor dopant for this purpose which would (unlike carbon) have a segregation coefficient less than unity. In the LEC growth of SI GaAs at Cominco/J.M., this was at first an accidental discovery when their source Ga or As contained trace amounts of zinc and/or of cadmium (which is also usable in the same manner). Cominco/J.M. went on to use this deliberately. Of course, J.M. is now retired from the business, and carbon doping of LEC grown GaAs is now known to be feasible continuously through growth with CO vapor in the atmosphere; but zinc or cadmium doping could be valuable for VZM growth where one is unable to make continuous adjustments to the atmosphere during growth.

I defer other reporting on the electrical samples from Crystal 4-88H until the data have been introduced for VZM Crystal 27-19N.

Report on Samples from Crystal 27-19N

Two thick polished slabs for optical measurements (carbon LVM and EL2), and two thin wafers from which electrical samples were to be prepared, were received from this crystal on August 16, 1991. The four sample wafer numbers were noted in Table I on page 13, and a summary of the room temperature properties is given in Table V, on page 24. In explaining that table and my estimation of this crystal, I shall begin again with reporting the measurements of carbon LVM absorption.

Figures 12 and 13, on pages 25 and 26, show traces of the transmittance through the mid-IR range $600 - 550 \text{ cm}^{-1}$ for Samples II-1 and II-31 of Crystal 27-19N. It was encouraging that both traces did show an extra little dip of transmittance centered on about 580 cm^{-1} , indicative of a modest but clearly apparent presence of C_{As} acceptors. Figures 14 and 15, on pages 27 and 28, show the region $590 - 570 \text{ cm}^{-1}$ in more detail. It is interesting that, for this crystal, the amount of carbon evident is just about as prominent for Sample II-31 as it is for Sample II-1 from much closer to the seed.

And so, on to the near-IR absorption, which one expects to arise predominantly from EL2 - mostly from photoionization of neutral EL2, but maybe to some extent from ionized EL2 photoneutralization. Figure 16 on page 29 shows the variation of absorption coefficient with $h\nu$ for Sample 27-19N-II-1. The general shape is as reported for numerous SI GaAs samples (as in Figs. 2, 10, and 11, and clearly arises mostly from the photoionization process. The needs of Table V are served adequately just by taking the value of α for a wavelength of $1.1 \mu\text{m}$ (i.e., for $h\nu = 1.127 \text{ eV}$), and this value $\alpha = 0.545 \text{ cm}^{-1}$ is inserted in the column of numbers in Table V; thus providing numbers for N^0 , N^+ , and $N_{EL2} = (N^0 + N^+)$ as indicated further down that column in Table V. The resulting number for N_{EL2} (between 7 and $8 \times 10^{15} \text{ cm}^{-3}$) is quite typical for VZM-grown GaAs when no special variations have been done in the way of an extra-rich As atmosphere to try and promote an enlargement of N_{EL2} . Indeed, in view of the rapid cooldown procedure used with Crystal 27-19N, it is something of a gratifying surprise that N_{EL2} is so "normal" in VZM-growth terms.

Figure 17, on page 30, shows the corresponding plot of $\alpha(h\nu)$ for Sample II-31, further up from the seed region of Crystal 27-19N. No dramatic increase of N_{EL2} and of its attendant absorption was to be expected here; and indeed the absorption spectral curves for Samples II-1 and II-31 of Crystal 27-19N are pretty darn similar.

[Text resumes on page 31]

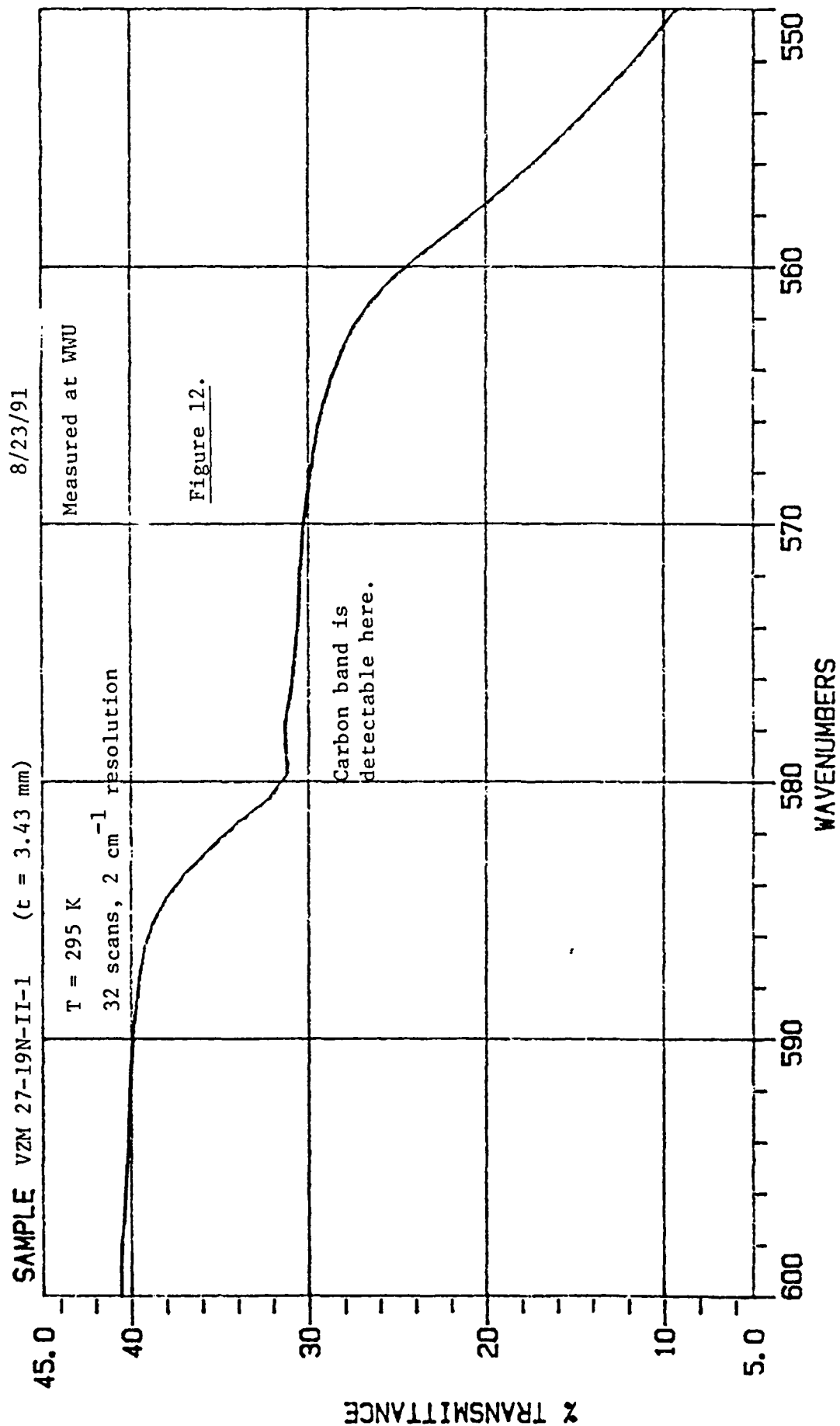
NRL Final Report 8/31/91 (Cont.)

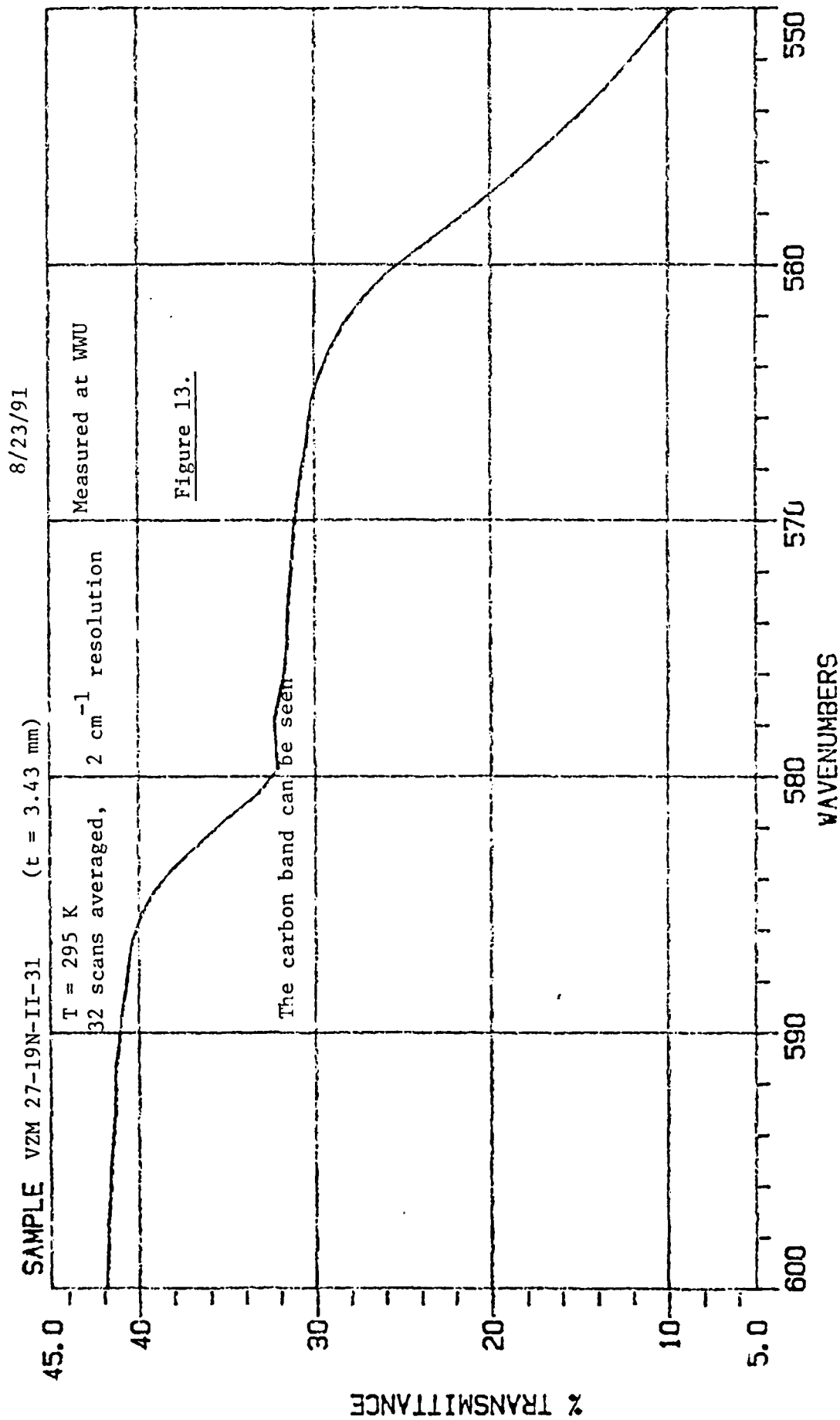
Table V. Summary of Room Temperature Properties for Samples from Undoped VZM-Grown GaAs Crystal 27-19N. Data Obtained August 1991.

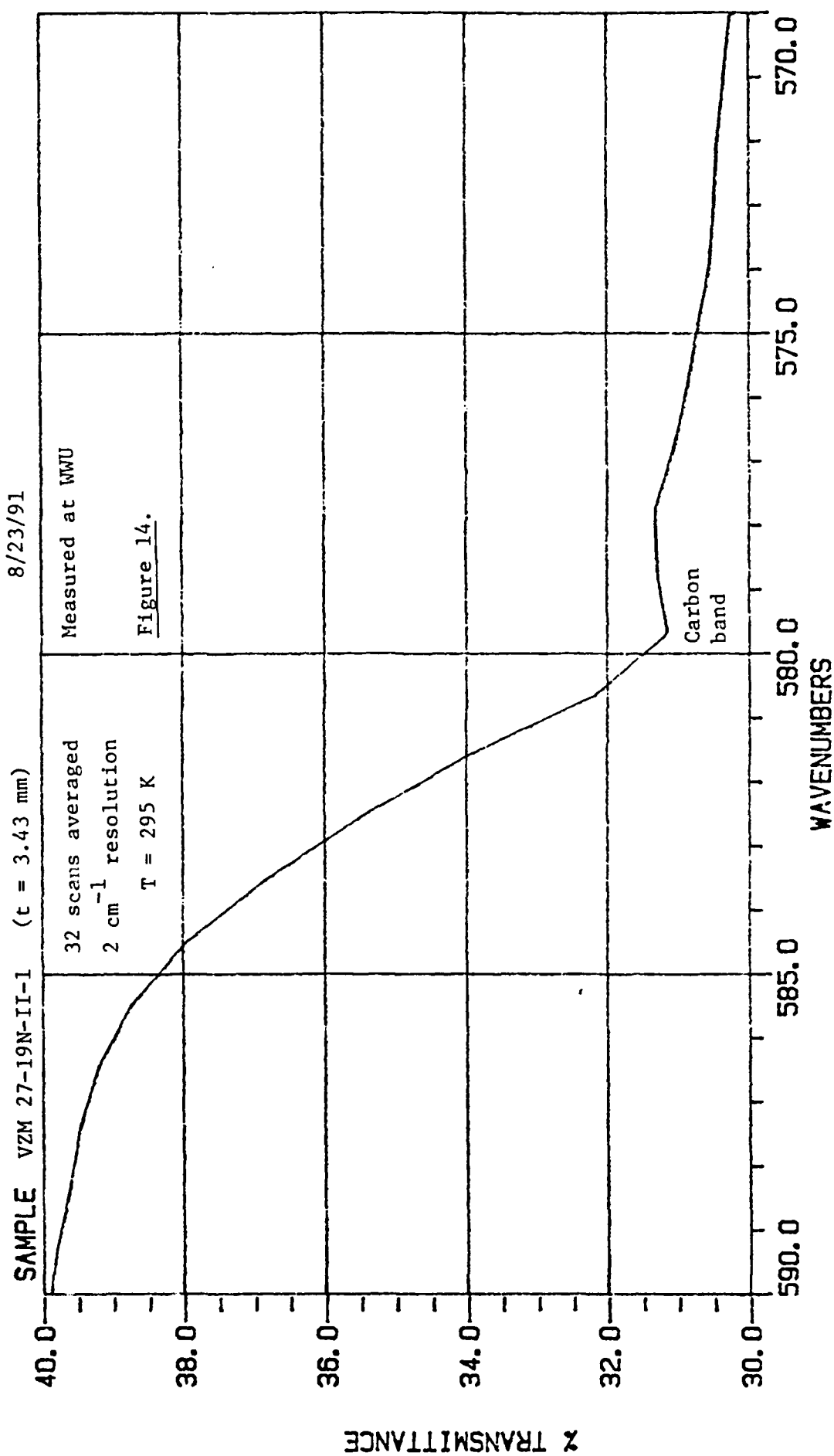
Note: Please see other tables and figures for the detailed data on this crystal, of which the table below gives a summary. As with most of the crystals described in this report, 27-19N was grown in Dr. Nordquist's VZM system at NRL. Atmosphere was 1 Atm argon.

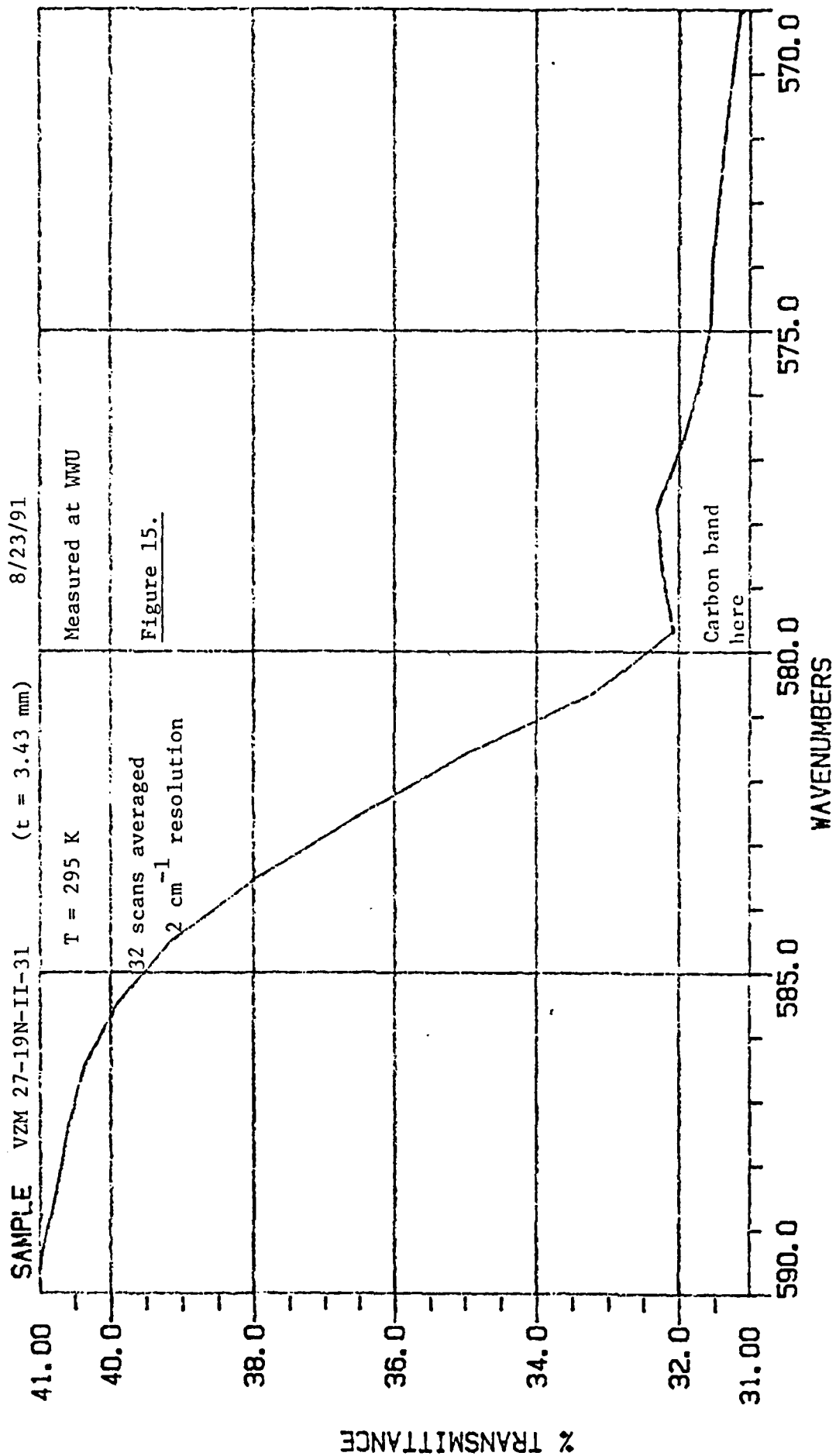
It is noteworthy that this crystal was given a rather brief period for liquid B₂O₃ to drain off after crystallization, followed by a rather rapid cooldown (160°C/hr) to ambient temperature.

Crystal	VZM 27-19N	
Optical Sample Slab	II-1	II-31
Electrical Data Wafer	II-5	II-25
300.0K Electrical Data:-		
Resistivity ρ (ohm cm)	1.56×10^8	1.39×10^8
Hall Coeff. R_H (cm ³ /Coulomb)	-8.54×10^{11}	-6.29×10^{11}
Ratio $z = (R_{Hi}/R_H)$	3.4	4.6
Ambipolar-corrected Electron Hall Mobility μ_{Hn} (cm ² /V s)	5497	4538
Ambipolar-corrected Electron Concentration n_o (cm ⁻³)	8.56×10^6	1.165×10^7
Ratio (n_o/n_i)	3.7	5.1
EL2 Ionized Fraction P_i	0.234	0.183
EL2 Concentrations (Neutral, Ionized, Total) Deduced from the above P_i values, combined with room temp (~295 K) near-IR absorption data:		
1.1 μ m Absorption Coeff. [α (cm ⁻¹)]	0.545	0.540
Deduced Neutral N^o (cm ⁻³)	5.8×10^{15}	5.9×10^{15}
" Ionized N^+ (cm ⁻³)	1.8×10^{15}	1.3×10^{15}
Thus, Total EL2 Concentration $N_{EL2} = (N^o + N^+) (cm^{-3})$	7.6×10^{15}	7.2×10^{15}
Evidence of carbon (C _{As}) from room temperature observations of the 580 cm ⁻¹ LVM absorption band.	Moderate, easily recognizable	For once, almost as much as in the other sample

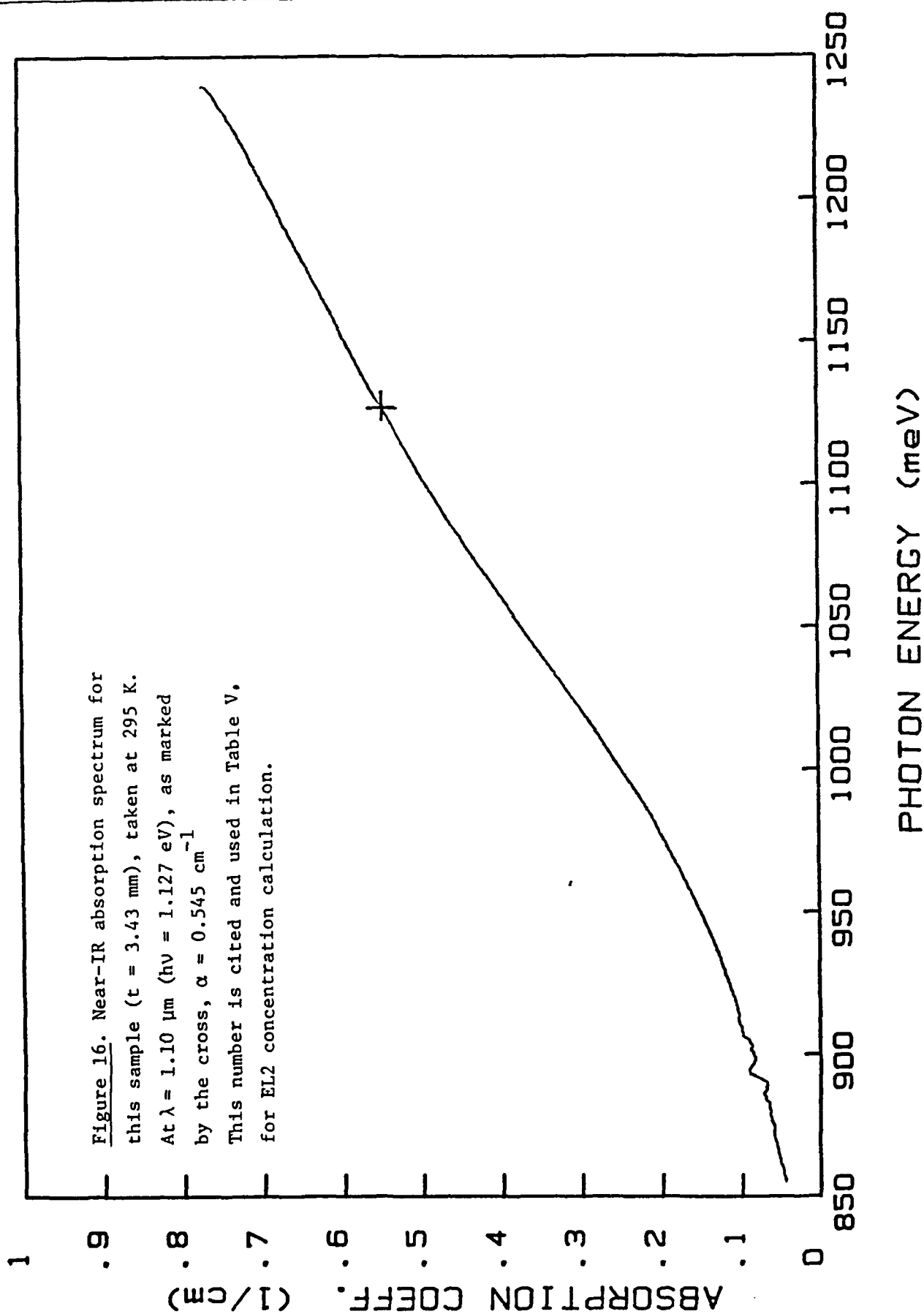






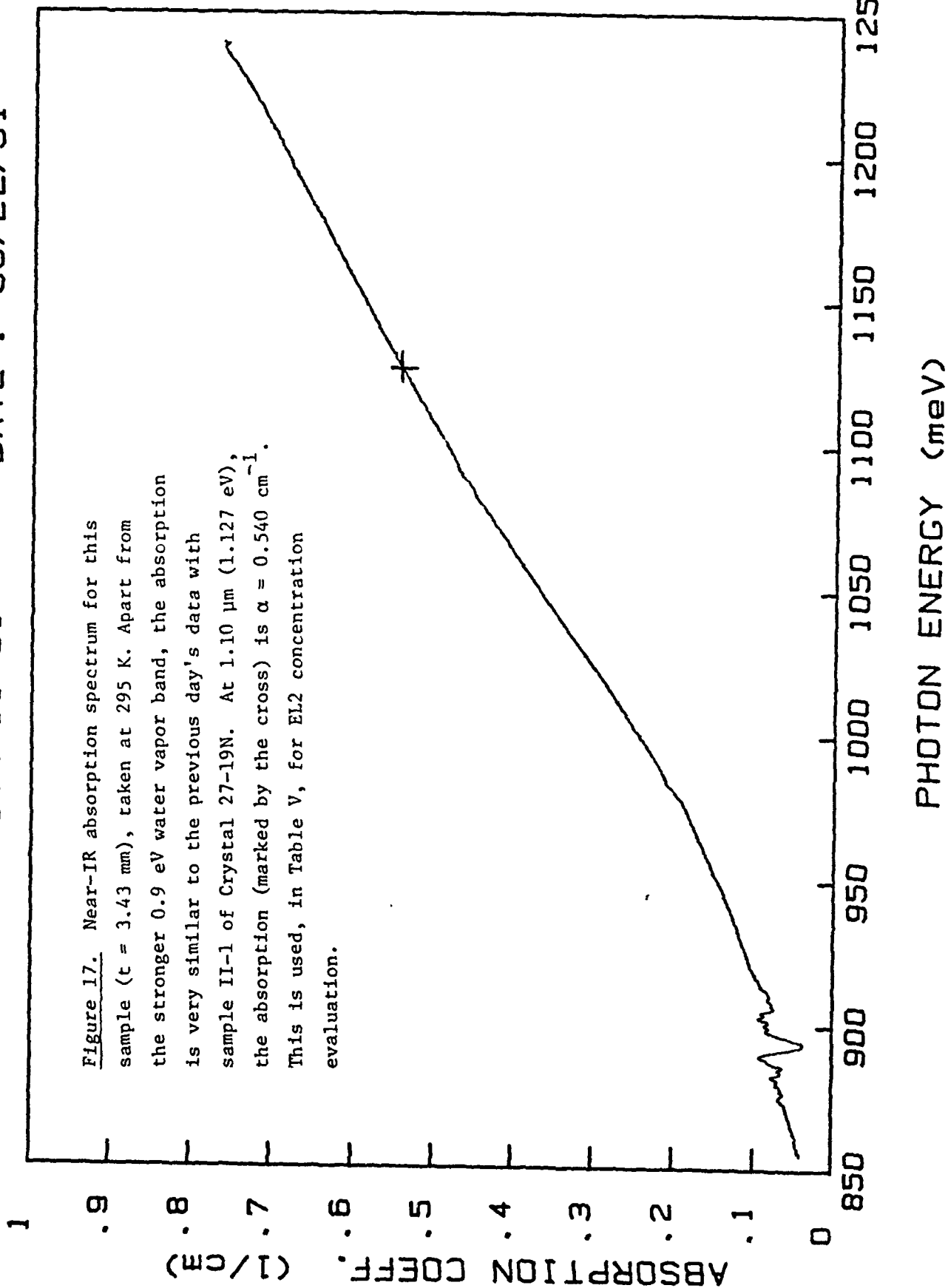


SAMPLE : V27-19N-II-1 DATE : 08/21/91



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SAMPLE : V27-19N-II-31 DATE : 08/22/91



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As with Figure 16, from the Sample II-31 curve in Figure 17, the absorption coefficient $\alpha = 0.540 \text{ cm}^{-1}$ at $\lambda = 1.10 \text{ } \mu\text{m}$ ($h\nu = 1.127 \text{ eV}$) is taken and inserted in the right-hand column of Table V. That is then used, in conjunction with the electrically-derived number for P_i , to determine the total EL2 concentration N_{EL2} , and its components N° and N^{+} .

And so the optical measurements have served their purposes, and it is time to report the full range of electrical measurements for which Table V had given only the 300 K numbers. Sample 27-19N-II-5 was measured at 300.0 K, and at four higher temperatures, with results tabulated in Table VI on page 32. The results for this sample were very pleasing, with a 300 K Hall mobility of nearly 5500 $\text{cm}^2/\text{V s}$, the mobility declining properly on warming. That the EL2 ionized fraction P_i should be as large as 23% was not altogether surprising in view of the noticeable carbon "dip" of transmittance in Figures 12 and 14. This amount of EL2 compensation resulted in an electron concentration only ~ 3.6 times larger than n_i (average over the temperature range), and an ambipolar correction for n_o and μ_{Hn} which is fairly small (less than 1%) but not negligible. In contrast to Sample 4-88H-III-9, here EL2 is in complete control.

The corresponding data for 300 K and four higher temperatures are tabulated in Table VII, on page 33, for Sample 27-19N-II-25, a sample evidently from higher up Crystal 27-19N. It will be remembered that the optical sample (II-31) from the upper part of that crystal had shown a gratifying amount of carbon (see Figs. 13 and 15), thus prior to any electrical measurements it had been reasonable to expect that there would be amply adequate EL2 compensation. And of course the 300 K numbers quoted in Table V gave that assurance. Now Table VII gives the data, raw and processed, for this and the other four temperatures. If it were always possible to ensure that the amount of carbon present declined only by $\sim 25\%$ from the beginning to well up a VZM crystal - as appears to have been the case for Crystal 27-19N - my concerns expressed on page 23 would be superfluous. No other kind of acceptor doping would be necessary. However, my experience based on measuring numerous VZM crystals is that Nature was kinder with 27-19N than usual. I still believe that some tiny doping to ensure adequate EL2 compensation as the crystal grows upwards, time after time, would bear more study.

I am now in a position to show the temperature-dependent results graphically, with data points extracted from Tables III, IV, VI, and VII, for the four electrical samples from both Crystals 4-88H and 27-19N. Figure 18, on page 34, shows the standard semilog plot of resistivity versus $(1/T)$. As forewarned, the slope is slightly smaller for Sample 4-88H-III-9, the sample for which control by EL2 partial compensation is inadequate.

Figure 19, on page 35, shows electron Hall mobility versus temperature for these four samples. The best mobility (and most healthy temperature dependence of mobility) is demonstrated by Sample 27-19N-II-5. Although the overall mobility is smaller for its brother sample 27-19N-II-25, that also has a healthy form of temperature dependence. The least desirable temperature dependence is of course shown by poor Sample 4-88H-III-9, with strong evidence there for interference by potential fluctuation barriers. Evidence for this is less strong with Sample 4-88H-II-5, but one can see that $\mu_{\text{Hn}}(T)$ is abnormally "flat". And, after all, Table III shows that P_i is only about 3% in this sample, so the risk of potential fluctuations is more than would have been the case if P_i had been only twice as large. As the reader can tell from my words, I am not in favor of having an unduly large P_i , but believe that one which is too small and fragile should be avoided if at all possible.

[Text resumes on page 37]

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Table VI. Electrical Data vs. Temperature for Sample 27-19N-II-5 ($t = 0.645$ mm)
from Undoped VZM-Grown GaAs Crystal 27-19N. (Data of 8/26/91)

Note: This was one of the series of crystals grown by Dr. Nordquist. For this one, a 1 Atm pressure of argon was maintained during growth, and the cooldown was rapid.

The first set of columns show the raw data, and n_o calculated from $(-r_{Hn}/eR_H)$. The second set of columns show n and Hn as adjusted for ambipolar conduction, the changes being small but not trivial. The third set of columns include the evaluation of EL2 ionized fraction, P_i .

TEMP. (K)	RESISTIVITY (OHM-CM)	HALL COEFFICIENT (CM ³ /COUL.)	HALL MOBILITY (CM ² /VS)	CARRIER CONCENTRATION (CM ⁻³)	1000/T (1/K)
300	1.559E+8	-8.538E+11	5475	8.626E+6	3.333
312.7	4.590E+7	-2.395E+11	5218	3.048E+7	3.197
324.7	1.562E+7	-7.782E+10	4982	9.306E+7	3.079
337.4	5.374E+6	-2.561E+10	4765	2.803E+8	2.963
349.9	2.038E+6	-9.316E+9	4571	7.639E+8	2.857

TEMP. (K)	RESISTIVITY (OHM-CM)	HALL COEFFICIENT (CM ³ /COUL.)	HALL MOBILITY (CM ² /VS)	CARRIER CONCENTRATION (CM ⁻³)	1000/T (1/K)	Z
300	1.559E+8	-8.538E+11	5497	8.558E+6	3.333	3.39
312.7	4.590E+7	-2.395E+11	5241	3.022E+7	3.197	3.29
324.7	1.562E+7	-7.782E+10	5005	9.223E+7	3.079	3.25
337.4	5.374E+6	-2.561E+10	4788	2.777E+8	2.963	3.22
349.9	2.038E+6	-9.316E+9	4594	7.565E+8	2.857	3.17

SAMPLE NAME = V27-19N-II-5

DATE = 08/26/91

AMBIPOLAR CORRECTION DATA

T (K)	Ei (mev)	Phi (mev)	Ni (cm ⁻³)	No (cm ⁻³)	Nd* (cm ⁻³)	Ed (mev)	Ef (mev)	Pi 1/(1+No/Nd*)
300	711.2	40.9	2.303E+6	8.558E+6	2.608E+6	44.1	74.9	.2336
312.7	708.4	42.7	8.338E+6	3.022E+7	8.990E+6	44.7	77.4	.2293
324.7	705.6	44.3	2.571E+7	9.223E+7	2.654E+7	45.2	80	.2234
337.4	702.7	46	7.785E+7	2.777E+8	7.689E+7	45.7	83	.2168
349.9	699.8	47.7	2.147E+8	7.565E+8	2.036E+8	46.1	85.7	.2121

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Table VII. Electrical Data vs. Temperature for Sample 27-19N-II-25 (t = 0.59 mm),
from Undoped VZM-Grown GaAs Crystal 27-19N. (Data of 8/27/91).

Note: The corresponding data for Sample II-5 of this crystal are in Table VI. The present sample, from higher up the VZM crystal, has smaller compensation of its EL2, but not by much ($P_i \approx 0.18$, compared with 0.23 in Sample II-5). Thus the ambipolar correction in the second and third set of columns is small (only ~0.5%) but not vanishingly so.

TEMP. (K)	RESISTIVITY (OHM-CM)	HALL COEFFICIENT (CM ³ /COUL.)	HALL MOBILITY (CM ² /VS)	CARRIER CONCENTRATION (CM ⁻³)	1000/T (1/K)
300	1.390E+8	-6.293E+11	4527	1.170E+7	3.333
313	3.933E+7	-1.706E+11	4338	4.278E+7	3.194
325.7	1.255E+7	-5.166E+10	4116	1.401E+8	3.07
338.4	4.393E+6	-1.763E+10	4012	4.069E+8	2.955
349.4	1.878E+6	-7.245E+9	3857	9.826E+8	2.862

TEMP. (K)	RESISTIVITY (OHM-CM)	HALL COEFFICIENT (CM ³ /COUL.)	HALL MOBILITY (CM ² /VS)	CARRIER CONCENTRATION (CM ⁻³)	1000/T (1/K)	Z
300	1.390E+8	-6.293E+11	4538	1.165E+7	3.333	4.6
313	3.933E+7	-1.706E+11	4349	4.257E+7	3.194	4.49
325.7	1.255E+7	-5.166E+10	4127	1.394E+8	3.07	4.47
338.4	4.393E+6	-1.763E+10	4024	4.046E+8	2.955	4.3
349.4	1.878E+6	-7.245E+9	3869	9.768E+8	2.862	4.24

SAMPLE NAME = V27-19N-II-25 DATE = 08/27/91

AMBIPOLAR CORRECTION DATA

T (K)	Ei (mev)	Phi (mev)	Ni (cm ⁻³)	No (cm ⁻³)	Nd* (cm ⁻³)	Ed (mev)	Ef (mev)	Pi 1/(1+No/Nd*)
300	711.2	40.9	2.303E+6	1.165E+7	2.608E+6	44.1	82.8	.183
313	708.3	42.7	8.584E+6	4.257E+7	9.246E+6	44.7	85.9	.1784
325.7	705.4	44.4	2.814E+7	1.394E+8	2.894E+7	45.2	89.3	.172
338.4	702.4	46.2	8.466E+7	4.046E+8	8.333E+7	45.7	91.8	.1708
349.4	699.9	47.7	2.064E+8	9.768E+8	1.961E+8	46.1	94.5	.1672

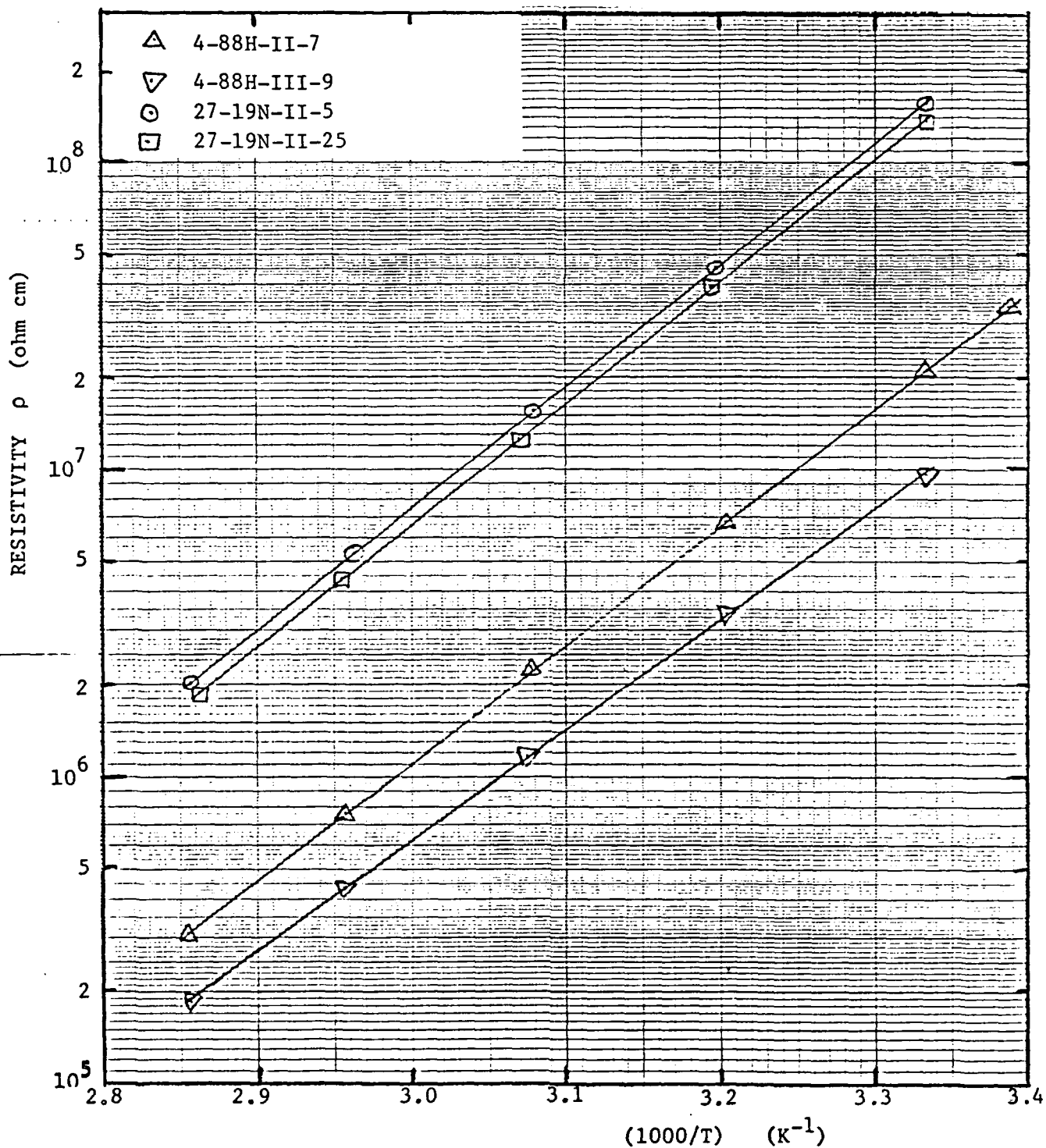


Figure 18. Semilog plot of resistivity vs. reciprocal temperature, for the two electrical samples of Crystal 4-88H (vide Tables III and IV), plus the electrical samples from Crystal 27-19N (vide Tables VI and VII. Note that the activation energy is slightly smaller for 4-88H-III-9 than for the other three samples, due to partial loss of EL2 control.

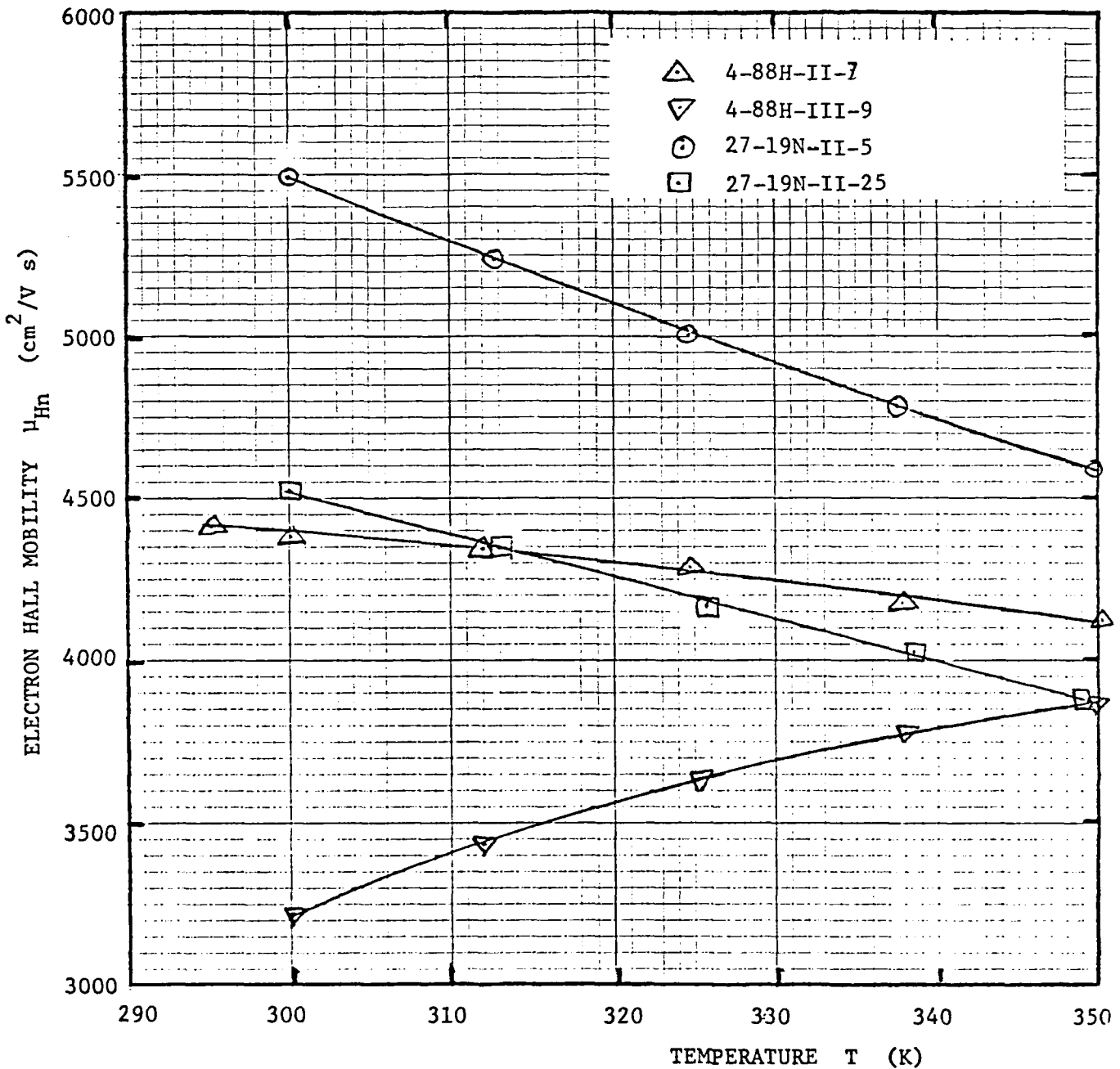


Figure 19. The Hall mobility of electrons, versus temperature, for the same four samples from Crystals 4-88H and 27-19N. The downwards progression of μ_{Hn} with rising temperature, seen in the two samples from Crystal 27-19N, is best consistent with phonon scattering (plus impurity scattering and other smaller effects). The flatter $\mu_{Hn}(T)$ for Sample 4-88H-II-7, and more especially the upwards trend of $\mu_{Hn}(T)$ for Sample 4-88H-III-9, suggest that microscopic potential barriers lower the mobility in this crystal, one for which the EL2 compensation is known to be weak.

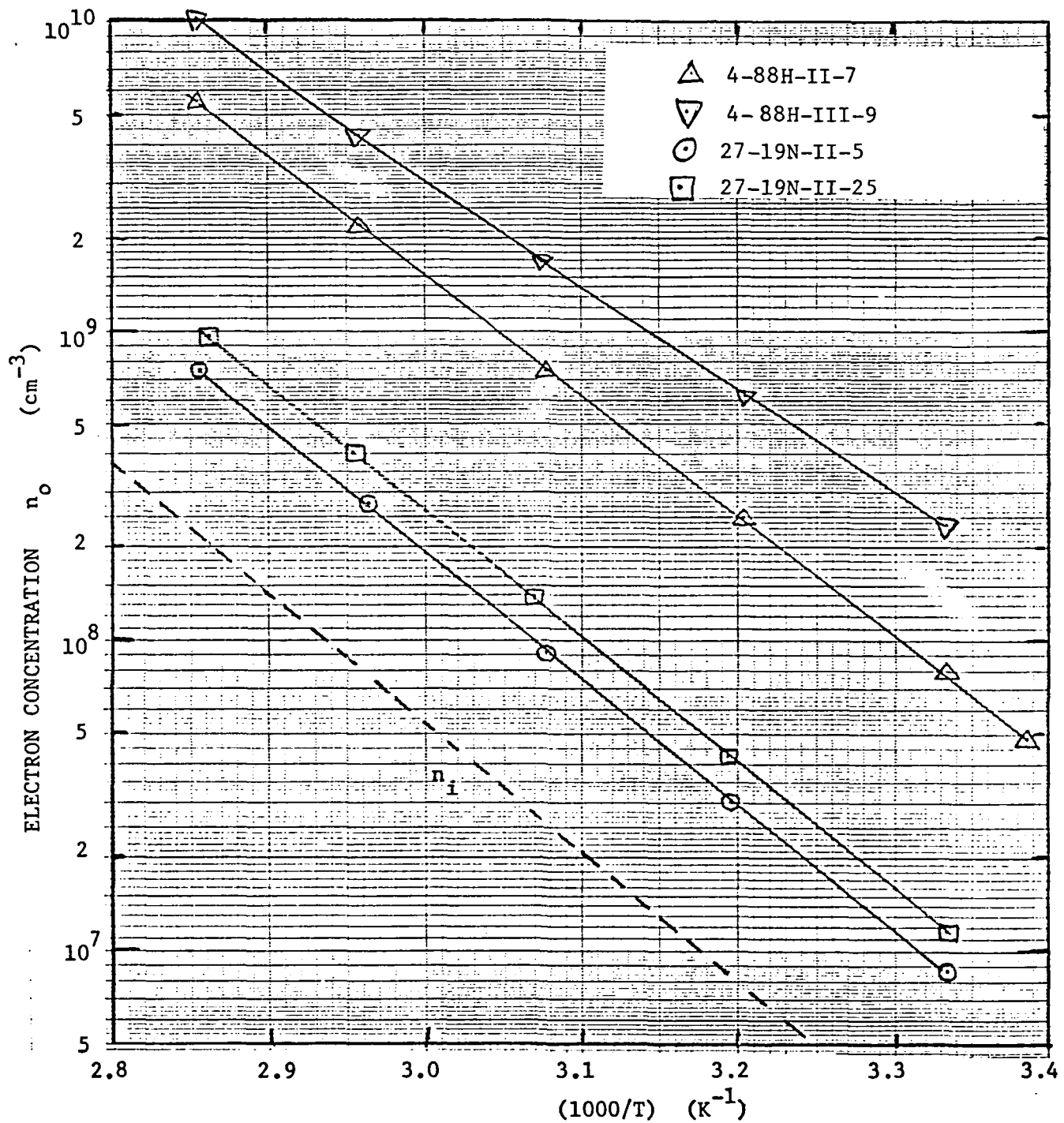


Figure 20. Electron concentration n_o vs. $(1000/T)$ for the same four samples from Crystals 4-88H and 27-19N, showing also the line for intrinsic $n_i(T)$ for comparison. Note that the uppermost data line (for Sample 4-88H-III-9) has a shallower slope - especially in the lower temperature part of the range - due to incomplete control of the Fermi energy by EL2 compensation.

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The final figure is Figure 20, on the preceding page (page 36), and this shows the conventional semilogarithmic plot of electron concentration versus $(1000/T)$ for these four electrical samples. Just for good measure, the track of $n_i(T)$ is also shown, reinforcing the notation in Table V that $n \approx 4n_i$ for Sample 27-19N-II-5, and that $n \approx 5n_i$ for its companion Sample II-25. Only for those two samples does the ambipolarⁱ correction procedure make any tangible shift in the numbers for n_o and μ_{Hn} .


Also visible in Figure 20 is the form of $n_o(T)$ for Sample 4-88H-III-9, which obviously has an activation energy too small to be fully controlled by EL2, this being especially obvious with the data for the lower part of the temperature range. If data collection had continued upwards in temperature, this sample would have moved closer towards full EL2 control. However, it is the region around 300 K which is of most concern for device substrate purposes.

Concluding Comments

The results obtained and reported during the past 18 months, including those given in detail as well as in summary form in this Final Technical Report, should provide encouragement to NRL staff concerning the viability of the VZM growth process for "undoped" GaAs. I am aware that other GaAs crystals have been VZM grown with substantial amounts of shallow dopant impurities (zone levelling), for conducting GaAs; but that has been outside the scope of our work here in characterizing the undoped and hopefully SI crystals. Especially in Dr. Nordquist's series of growth experiments with variations in the Ga-As ratio, the use of an argon atmosphere overpressure, the comparison of slow cool with rapid cool-down, etc., there have been a number of variables which have needed, and continue to need, characterization support.

I can state honestly that the need for this type of characterization is still present, and shall hope to be able to work further with NRL on reduction of VZM growth to as near to an exact science as possible. As a melt-growth method which can produce GaAs of small dislocation density, this is highly attractive as a method for provision of low-EPD substrates for optoelectronic applications. In that type of application, where the lasers, optical detectors, and associated electronic components are all fabricated in a series of epilayers, the actual substrate resistivity needs only to be adequately large; and the electron mobility in that substrate does not need to be high. Nevertheless, along with preservation of the low EPD, one still would like to know that undoped GaAs grown by VZM was of excellent electronic quality. Further experimentation towards that goal is to be recommended.

Sincerely yours,


John S. Blakemore